

APPENDIX A

PROGRAM TEST METHODS

Appendix A.1

Ontario-Hydro Mercury Method

Standard Test Method for Elemental, Oxidized, Particle-Bound, and Total Mercury in Flue Gas Generated from Coal-Fired Stationary Sources (Ontario Hydro Method)¹

1. Scope

1.1 This method applies to the determination of elemental, oxidized, particle-bound, and total mercury emissions from coal-fired stationary sources.

1.2 This method is applicable to elemental, oxidized, particle-bound, and total mercury concentrations ranging from approximately 0.5 to 100 µg/dscm.

1.3 This method describes equipment and procedures for obtaining samples from effluent ducts and stacks, equipment and procedures for laboratory analysis, and procedures for calculating results.

1.4 This method is applicable for sampling elemental, oxidized, and particle-bound mercury at the inlet and outlet of emission control devices and for calculating control device mercury collection efficiency.

1.5 Method applicability is limited to flue gas stream temperatures within the thermal stability range of the sampling probe and filter components.

1.6 The values stated in SI units are to be regarded as the standard. The values in parentheses are for information only.

1.7 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

1.8 This standard assumes users are familiar with EPA stack-gas sampling procedures as stated in EPA Methods 1-4, Method 5, and Method 17.

2. Referenced Documents

- 2.1 *ASTM Standards:*
D 1193 Specification for Reagent Water²

¹ This test method is under the jurisdiction of ASTM Committee D-22 on Sampling and Analysis of Atmospheres and is the direct responsibility of Subcommittee D22.03 on Ambient Atmospheres and Source Emissions.

² *Annual Book of ASTM Standards*, Vol. 11.01.

D1356 Definitions of Terms Relating to Atmospheric Sampling and Analysis³
D 2986 Evaluation of Air-Assay Media by the Monodisperse DOP (Diocetyl Phthalate) Smoke Test³
D 3154 Test Method for Average Velocity in a Duct (Pitot Tube Method)³
D 3685 Particulates Independently or for Particulates and Collected Residue Simultaneously in Stack Gases³
E 1 Specification for ASTM Thermometers⁴

*2.2 Other Standards:*⁵

EPA Method 1 – Sample and Velocity Traverses for Stationary Sources
EPA Method 2 – Determination of Stack Gas Velocity and Volumetric Flow Rate (Type S Pitot Tube)
EPA Method 3 – Gas Analysis for the Determination of Dry Molecular Weight
EPA Method 4 – Determination of Moisture Content in Stack Gases
EPA Method 5 – Determination of Particulate Emissions from Stationary Sources
EPA Method 12 – Determination of Inorganic Lead Emissions from Stationary Sources
EPA Method 17 – Determination of Particulate Emissions from Stationary Sources (In-Stack Filtration Method)
EPA Method 29 – Determination of Metals Emissions from Stationary Sources
EPA Method 101A – Determination of Particle-Bound and Gaseous Mercury Emissions from Sewage Sludge Incinerators
EPA Method 301 – Field Validation of Pollutant Measurement Methods from Various Waste Media
EPA SW 846 7470 – Mercury in Liquid Waste – Manual Cold Vapor Technique
EPA Water and Waste 600/4-79-020 – Methods for Chemical Analysis of Water and Wastes

3. Terminology

3.1 Definitions other than those given below in Sections 3.2, 3.3, and 3.4 are listed in ASTM D 1356.

3.2 *Definitions of Terms specific to the standard:*

3.2.1 *elemental mercury*—mercury in its zero oxidation state, Hg⁰.

³ *Annual Book of ASTM Standards*, Vol. 11.03.

⁴ *Annual Book of ASTM Standards*, Vol 14.02.

⁵ Available from the U.S. Environmental Protection Agency's Emission Measurement Technical Information Center or Code of Federal Regulations (40 CFR Part 60, Appendix A or 40 CFR Part 61, Appendix B).

3.2.2 *oxidized mercury*—mercury in its mercurous or mercuric oxidation states: Hg_2^{2+} and Hg^{2+} , respectively.

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3.2.3 *elemental mercury catch*—mercury collected in the acidified hydrogen peroxide ($\text{HNO}_3\text{--H}_2\text{O}_2$) and potassium permanganate ($\text{H}_2\text{SO}_4\text{--KMnO}_4$) impinger solutions employed in this method. This is gaseous Hg^0 .

3.2.4 *oxidized mercury catch*—mercury collected in the aqueous potassium chloride (KCl) impinger solution employed in this method. This is gaseous Hg^{2+} .

3.2.5 *particle-bound mercury catch*—mercury associated with the particulate matter collected in the front half of the sampling train.

3.2.6 *front half of the sampling train*—all mercury collected on and upstream of the sample filter.

3.2.7 *total mercury*—all mercury (solid-bound, liquid, or gaseous) however generated or entrained in the flue gas stream (i.e., summation of elemental, oxidized, and particle-bound mercury).

3.3 Symbols:

A	= cross-sectional area of stack, m^2 (ft^2)
B_{ws}	= water vapor in the gas stream, proportion by volume
ΔH	= average pressure differential across the orifice meter, kPa (in. H_2O)
Hg_{ash}	= concentration of mercury in sample filter ash, $\mu\text{g/g}$
Hg^{tp}	= concentration of particle-bound mercury, $\mu\text{g/dscm}$
Hg^0	= concentration of elemental mercury, $\mu\text{g/dscm}$
Hg^{2+}	= concentration of oxidized mercury, $\mu\text{g/dscm}$
IR	= instrument reading from mercury analyzer, $\mu\text{g/L}$
L_p	= leakage rate observed during the posttest leak check, m^3/min (cfm)
L_a	= maximum acceptable leakage rate
M_s	= molecular weight of stack gas, wet basis, g/g-mole (lb/lb-mole)
M_w	= molecular weight of water, 18.0 g/g-mole (18.0 lb/lb-mole)
N	= Normal conditions, defined as 0°C and 1 atmosphere pressure (in the U.S. N and standard conditions are the same in SI units)
P_{bar}	= barometric pressure at the sampling site, kPa (in. Hg)
P_s	= absolute stack gas pressure, kPa (in. Hg)
P_{std}	= standard absolute pressure, 101.3 kPa (29.92 in. Hg)
R	= ideal gas constant, $0.008314 \text{ kPa}\cdot\text{m}^3/\text{K}\cdot\text{g-mole}$ ($21.85 \text{ in. Hg}\cdot\text{ft}^3/^\circ\text{R}\cdot\text{lb-mole}$)
T_m	= absolute average dry gas meter temperature, K ($^\circ\text{R}$)
T_s	= absolute stack temperature, K ($^\circ\text{R}$)
T_{std}	= standard absolute temperature, 293 K (528°R)
V_D	= total digested volume, mL
V_m	= volume of gas sample as measured by dry gas meter, dcm (dscf)

$V_{m(std)}$ = volume of gas sample measured by the dry gas meter, corrected to standard conditions, dscm (dscf)
 $V_{w(std)}$ = volume of water vapor in the gas sample, corrected to standard conditions, scm (scf)
 W_{ash} = total mass of ash on sample filter, g
 W_{lc} = total weight of liquid collected in impingers and silica gel, g (lb)
 Y = dry gas meter calibration factor
 θ = total sampling time, min
 θ_1 = sampling time interval, from the beginning of a run until the first component change, min

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4. Summary of Test Method

4.1 A sample is withdrawn from the flue gas stream isokinetically through a probe/filter system, maintained at 120°C or the flue gas temperature, whichever is greater, followed by a series of impingers in an ice bath. Particle-bound mercury is collected in the front half of the sampling train. Oxidized mercury is collected in impingers containing a chilled aqueous potassium chloride solution. Elemental mercury is collected in subsequent impingers (one impinger containing a chilled aqueous acidic solution of hydrogen peroxide and three impingers containing chilled aqueous acidic solutions of potassium permanganate). Samples are recovered, digested, and then analyzed for mercury using cold-vapor atomic absorption (CVAAS) or fluorescence spectroscopy (CVAFS).

5. Significance and Use

5.1 The measurement of particle-bound, oxidized, elemental, and total mercury in stationary-source flue gases provides data that can be used for dispersion modeling, deposition evaluation, human health and environmental impact assessments, emission reporting, compliance determinations, etc. Particle-bound, oxidized, and elemental mercury measurements before and after control devices may be necessary for optimizing and evaluating the mercury removal efficiency of emission control technologies.

6. Interferences

There are no known interferences, but certain biases may be encountered (See Section 16).

7. Apparatus

7.1 *Sampling Train*—similar to ASTM D 3685, EPA Method 5/EPA Method 17 and EPA Method 29 trains, as illustrated in Fig. 1.

7.1.1 *Probe Nozzle (Probe Tip)*—Glass nozzles are required unless alternate nozzles are constructed of materials that are free from contamination and will not interact with the sample. Probe fittings constructed of polytetrafluoroethylene (PTFE), polypropylene, etc., are required instead of metal fittings to prevent contamination.

7.1.2. *Probe Liner*—If the sample train is to be in EPA Method 5 configuration (out-of-stack filtration), the probe liner must be constructed of quartz or borosilicate glass. If an EPA Method 17 (in-stack filtration) sampling configuration is used, the probe/probe liner may be constructed of borosilicate glass, quartz or, depending on the flue gas temperature, PTFE.

7.1.3 *Pitot Tube*—Type S pitot tube. Refer to Section 2.2 of EPA Method 2 for a description.

7.1.4 *Differential Pressure Gauges*—inclined manometers or equivalent devices. Refer to Section 2.1 of EPA Method 2 for a description.

7.1.5 *Filter Holder* — constructed of borosilicate glass or PTFE-coated stainless steel with a PTFE filter support or other nonmetallic, noncontaminating support. Do not use a glass frit or stainless steel wire screen. A silicone rubber or PTFE gasket, designed to provide a positive seal against leakage from outside or around the filter, may be used.

7.1.6 *Connecting Umbilical Tube*—heated PTFE tubing. This tube must be heated to a minimum of 120°C to help prevent water and acid condensation. (The umbilical tube is defined as any tubing longer than 0.5 m that connects the filter holder to the impinger train).

7.1.7 *Probe and Filter Heating System*

7.1.7.1 *EPA Method 5 Configuration*—For EPA Method 5 configuration, the temperature of the flue gas, sample probe, and the exit of the sample filter must be monitored using temperature sensors capable of measuring temperature to within 3°C (5.4°F). The heating system must be capable of maintaining the sample gas temperature of the probe and exit of the sample filter to within $\pm 15^\circ\text{C}$ ($\pm 27^\circ\text{F}$) of the flue gas temperature. Regardless of the flue gas temperature, to prevent water and acid condensation, at no time must the probe temperature, sample filter exit gas temperature, or the temperature of the connecting umbilical cord be less than 120°C.

7.1.7.2 *EPA Method 17 Configuration*—For EPA Method 17 configuration, the sample filter is located in the duct and, therefore, naturally maintained at the flue gas temperature. The heating system is only required to maintain the probe and connecting umbilical cord to at least 120°C. If the flue gas temperature is less than 120°C, then EPA Method 5 configuration must be used.

7.1.8 *Condensing/Absorbing System*—consists of eight impingers immersed in an ice bath and connected in series with leak-free ground glass fittings or other noncontaminating leak-free fittings. (At no time is silicon grease or other greases to be used for this method). The first, second, fourth, fifth, sixth, and eighth impingers are of the Greenburg-Smith design modified by replacing the standard tip with a 1.3-cm (0.5-in.)-ID straight glass tube extending to about 1.3 cm (0.5 in.) from the bottom of the flask. The third and seventh impingers are also Greenburg-Smith design, but with the standard tip including the glass impinging plate. The first, second, and third impingers contain aqueous 1 N potassium chloride (KCl) solution. The fourth impinger contains

an aqueous solution of 5%^{V/V} nitric acid (HNO₃) and 10%^{V/V} hydrogen peroxide (H₂O₂). The fifth, sixth, and seventh impingers contain an aqueous solution of 4%^{W/V} potassium permanganate (KMnO₄) and 10%^{V/V} sulfuric acid (H₂SO₄). The last impinger contains silica gel or an equivalent desiccant. Refer to Note 1.

Note 1—When flue gas streams are sampled with high moisture content (>20%), additional steps must be taken to eliminate carryover of impinger contents from one sample type to the next. These steps must include use of oversized impinger(s) or use of an empty impinger between the KCl and HNO₃–H₂O₂. If a dry impinger is used, it must be rinsed as discussed in Section 13.2 of this method and the rinse added to the preceding impinger.

7.1.9 *Metering System*—vacuum gauge, leak-free pump, thermometers capable of measuring temperature to within 3°C (5.4°F), and a dry gas meter or controlled orifice capable of measuring volume to within 2%.

7.1.10 *Barometer*—barometer capable of measuring atmospheric pressure to within 0.33 kPa (0.1 in. Hg). In many cases, the barometric reading may be obtained from a nearby National Weather Service station, in which case, the station value (which is the absolute barometric pressure) shall be requested. An adjustment for elevation differences between the weather station and sampling point shall be applied at a rate of negative 0.33 kPa (0.1 in. Hg) per 30 m (100 ft) elevation increase or vice versa for elevation decrease.

7.1.11 *Gas Density Determination Equipment*—temperature sensor and pressure gauge, as described in Section 2.3 and 2.4 of EPA Method 2. The temperature sensor shall, preferably, be permanently attached to the pitot tube or sampling probe in a fixed configuration, such that the sensor tip extends beyond the leading edge of the probe sheath and does not touch any metal. Alternative temperature sensor configurations are described in Section 2.1.10 of EPA Method 5. If necessary, a gas analyzer can be used to determine dry molecule weight of the gas (refer to EPA Method 3).

7.2 Digestion Apparatus

7.2.1 *Dry Block Heater or Hot Water Bath*—a heater capable of maintaining a temperature of 95°C is required for digestion of samples, similar to that described in EPA SW846 Method 7470.

7.2.2 Ice Bath

7.2.3 *Digestion Flasks*—Use 50- to 70-mL tubes or flasks with screw caps that will fit a dry block heater. For a water bath, 300-mL biological oxygen demand bottles for SW846 Method 7470 are to be used. In addition, borosilicate glass test tubes, 35- to 50-mL volume, with rack are needed.

7.2.4 *Microwave or Convection Oven and PTFE Digestion Vessels*—120 mL, or equivalent digestion vessels with caps equipped with pressure relief valves for the dissolution of

ash, along with a capping station or the equivalent to seal the digestion vessel caps. Use a vented microwave or convection oven for heating. In addition, polymethylpentene (PMP) or equivalent volumetric flasks are recommended for the digested ash solutions.

7.3 *Analytical Equipment*—dedicated mercury analyzer or equivalent apparatus for the analysis of mercury via CVAAS. Alternatively, CVAFS may be used. CVAAS is a method based on the absorption of radiation at 253.7 nm by mercury vapor. The mercury is reduced to the elemental state and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrometer. Absorbency is measured as a function of mercury concentration. A soda-lime trap and a magnesium perchlorate trap must be used to precondition the gas before it enters the absorption cell.

8. Reagents and Materials

8.1 *Purity of Reagents*—Reagent-grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.⁶ Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

8.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type II in ASTM Specification D 1193.

8.3 *Reagents:*

8.3.1 *Boric Acid (H_3BO_3)*—purified reagent grade.

8.3.2 *Hydrochloric Acid (HCl)*—trace metal-grade concentrated hydrochloric acid, with a specific gravity of 1.18.

8.3.3 *Hydrofluoric Acid (HF)*—concentrated hydrofluoric acid, 48%–50%.

8.3.4 *Hydrogen Peroxide (H_2O_2)*—30%^{V/V} hydrogen peroxide.

8.3.5 *Hydroxylamine Sulfate ($NH_2OH \cdot H_2SO_4$)*—solid.

8.3.6 *Mercury Standard Solution*—a certified (1000 µg/mL) mercury standard.

8.3.7 *Nitric Acid (HNO_3)*—trace metal-grade concentrated nitric acid with a specific gravity of 1.42.

⁶ “Reagent Chemicals, American Chemical Society Specifications,” Am. Chemical Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see “Reagent Chemicals and Standards,” by Joseph Rosin, D. Van Nostrand Co., Inc., New York, NY, and the “United States Pharmacopeia.”

8.3.8 *Potassium Chloride (KCl)*—solid.

8.3.9 *Potassium Permanganate (KMnO₄)*—solid.

8.3.10 *Potassium Persulfate (K₂S₂O₈)*—solid.

8.3.11 *Stannous Chloride (SnCl₂ · 2H₂O)*—solid.

8.3.12 *Sulfuric Acid (H₂SO₄)*—trace metal-grade concentrated sulfuric acid, with a specific gravity of 1.84.

8.4 *Materials:*

8.4.1 *Indicating Silica Gel*—with a size of 6–16 mesh.

8.4.2 *Crushed or Cubed Ice.*

8.4.3 *Sample Filters*—quartz fiber filters, without organic binders, exhibiting at least 99.95% efficiency (<0.05% penetration) for 0.3-μm dioctyl phthalate smoke particles and containing less than 0.2 μg/m² of mercury. Test data provided by filter manufacturers and suppliers stating filter efficiency and mercury content are acceptable. However, if no such results are available, determine filter efficiency using ASTM Test Method D 2986, and analyze filter blanks for mercury prior to emission testing. Filter material must be unreactive to sulfur dioxide (SO₂) or sulfur trioxide (SO₃).⁷

8.4.4 *Filter Papers*—for filtration of digested samples. The filter paper must have a particle retention of >20 μm and filtration speed of >12 sec.

8.4.5 *Nitrogen Gas (N₂)*—carrier gas of at least 99.998% purity. Alternatively, argon gas may be used.

8.4.6 *Soda Lime*—indicating 4- to 8-mesh absorbent for trapping carbon dioxide.

8.4.7 *Sample Containers*—glass with PTFE-lined lids.

8.5 *Sampling Reagents*

8.5.1 *KCl Absorbing Solution (1 mol/L)*—Dissolve 74.56 g of KCl in 500 mL of reagent water in a 1000-mL volumetric flask, swirl to mix, and dilute to volume with water. Mix well. A new batch of solution must be made prior to each field test.

⁷ Felix, L.G.; Clinard, G.I.; Lacey, G.E.; McCain, J.D. "Inertial Cascade Impactor Substrate Media for Flue Gas Sampling," U.S. Environmental Protection Agency, Research Triangle Park, NC 27711, Publication No. EPA-600/7-77-060; June 1977, 83 p.

8.5.2 *HNO₃-H₂O₂ Absorbing Solution* (5%^{v/v} HNO₃, 10%^{v/v} H₂O₂)—Add slowly, with stirring, 50 mL of concentrated HNO₃ to a 1000-mL volumetric flask containing approximately 500 mL of water, and then add carefully, with stirring, 333 mL of 30%^{v/v} H₂O₂. Dilute to volume with water. Mix well. A new batch of solution must be made prior to each field test.

8.5.3 *H₂SO₄-KMnO₄ Absorbing Solution* (4%^{w/v} KMnO₄, 10%^{v/v} H₂SO₄)—Mix carefully, with stirring, 100 mL of concentrated H₂SO₄ into approximately 800 mL of water. When mixing, be sure to follow standard acid to water addition procedures and safety precautions associated with strong acids. Then add water, with stirring, to make 1 L. This solution is 10%^{v/v} H₂SO₄. Dissolve, with stirring, 40 g of KMnO₄ into 10%^{v/v} H₂SO₄, and add 10%^{v/v} H₂SO₄, with stirring, to make 1 L. (Warning—See 9.1.1). H₂SO₄-KMnO₄ absorbing Solution must be made daily.

8.6 *Rinse Solutions for Sample Train*

8.6.1 *0.1 N HNO₃ Solution*—A certified reagent grade 0.1 N HNO₃ solution can be purchased directly or can be made by slowly adding 12.5 mL of concentrated HNO₃ to a 2000-mL volumetric flask containing approximately 500 mL of water, then diluting with water to volume.

8.6.2 *10%^{w/v} HNO₃ Solution*—Mix carefully, with stirring, 100 mL of concentrated HNO₃ into approximately 800 mL of water. When mixing, be sure to follow standard acid to water addition procedures and safety precautions associated with strong acids. Then add water, with stirring, to make 1 L.

8.6.3 *10%^{w/v} Hydroxylamine Sulfate*—Add 100 g hydroxylamine sulfate to a 1000-mL volumetric flask containing approximately 500 mL of water. After the hydroxylamine sulfate has been dissolved, dilute with water to volume.

8.7 *Sample Digestion Reagents:*

8.7.1 *Boric Acid Solution* (4%^{w/v})—Dissolve 4 g H₃BO₃ in water, and dilute to 100 mL.

8.7.2 *Aqua Regia (HCl:HNO₃ 3:1)*—Add 3 parts concentrated HCl to 1 part concentrated HNO₃. Note that this should be made up in advance and allowed to form a dark orange color. This mixture should be loosely capped, as pressure will build as gases form.

8.7.3 *Saturated Potassium Permanganate Solution* (5%^{w/v})—Mix 5 g KMnO₄ into water, dilute to 100 mL, and stir vigorously.

8.7.4 *Potassium Persulfate Solution* (5%^{w/v})—Dissolve 5 g K₂S₂O₈ in water, and dilute to 100 mL.

8.8 *Analytical Reagents:*

8.8.1 *Hydrochloric Acid Solution (10%^V/_V)*—Add 100 mL concentrated HCl to water, and dilute to 1 L. Be sure to follow all safety precautions for using strong acids. **DRAFT**

8.8.2 *Stannous Chloride Solution (10%^W/_V)*—Dissolve 100 g in 10%^V/_V HCl, and dilute with 10%^V/_V HCl to 1 L. Difficulty in dissolving the stannous chloride can be overcome by dissolving in a more concentrated HCl solution (such as 100 mL of 50%^V/_V HCl) and diluting to 1 L with water. Note that care must be taken when adding water to a strong acid solution. Add a lump of mossy tin (~0.5 g) to this solution.

8.9 *Mercury Standards:*

8.9.1 *10 mg/L Hg Stock Solution*—Dilute 1 mL of 1000 mg/L Hg standard solution to 100 mL with 10%^V/_V HCl.

8.9.2 *100 µg/L Hg Stock Solution*—Dilute 1 mL of 10 mg/L Hg stock solution to 100 mL with 10%^V/_V HCl.

8.9.3 *Working Hg Standards*—Prepare working standards of 1.0, 5.0, 10.0, and 20.0 µg/L Hg from the 100-µg/L stock solution by diluting 1, 5, 10, and 20 mL each to 100 mL with 10%^V/_V HCl.

Note 1—If samples to be analyzed are less than 1.0 µg/L Hg, working standards should be prepared at 0.05, 0.1, 0.5, and 1.0 µg/L Hg from a 10-µg/L Hg standard solution.

8.9.4 *Quality Control Standard (QC)*—A quality control standard is prepared from a separate Hg standard solution. The QC standard should be prepared at a concentration of approximately one-half the calibration range.

8.10 *Glassware Cleaning Reagents*—Prior to any fieldwork, all glassware should be cleaned according to the guidelines outlined in EPA Water and Waste 600/4-79-020, Section 4, pages 4–5.

9. **Hazards**

9.1 *Warning:*

9.1.1 Pressure may build up in the solution storage bottle because of a potential reaction between potassium permanganate and acid. Therefore, these bottles should not be fully filled and should be vented to relieve excess pressure and prevent explosion. Venting must be in a manner that will not allow contamination of the solution.

9.1.2 Hazards to personnel exist in the operation of the cold-vapor atomic absorption spectrophotometer. Refer to the manufacturer's instruction manual before operating the instrument.

9.1.3 Sample digestion with hot concentrated acids creates a safety problem. Observe appropriate laboratory procedures for working with concentrated acids.

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9.2 *Precaution:*

9.2.1 The determination of microquantities of mercury species requires meticulous attention to detail. Good precision is generally unattainable without a high level of experience with stack-sampling procedures. Precision may be improved by knowledge of, and close adherence to, the suggestions that follow.

9.2.1.1 All glassware used in the method must be cleaned thoroughly prior to use in the field, as described in Section 8.10 of this method.

9.2.1.2 Use the same reagents and solutions in the same quantities for a group of determinations and the corresponding solution blank. When a new reagent is prepared or a new stock of filters is used, a new blank must be taken and analyzed.

10. Sampling

10.1 *Preparation for Test:*

10.1.1 *Preliminary Stack Measurements*—Select the sampling site, and determine the number of sampling points, stack pressure, temperature, moisture, dry molecular weight, and range of velocity head in accordance with procedures of ASTM Test Method D 3154 or EPA Methods 1 through 4.

10.1.2 Select the correct nozzle diameter to maintain isokinetic sampling rates based on the range of velocity heads determined in 10.1.1.

10.1.3 Ensure that the proper differential pressure gauge is selected for the range of velocity heads (refer to EPA Method 2, Section 2.2).

10.1.4 It is suggested that an EPA Method 17 configuration be used; however, if an EPA Method 5 setup is to be used, then select a suitable probe length such that all traverse points can be sampled. Consider sampling from opposite sides of the stack to minimize probe length when a large duct or stack is sampled.

10.1.5 *Sampling Time and Volume*—The total sampling time for this method should be at least 2 but not more than 3 hours. Use a nozzle size that will guarantee an isokinetic gas sample volume between 1.0 dry cubic meters corrected to standard conditions (dscm) and 2.5 dscm. If traverse sampling is done (necessary for sampling at electric utilities), use the same points for sampling that were used for the velocity traverse as stated in Section 10.1.1 of this method. Each traverse point must be sampled for a minimum of 5 minutes.

11. Preparation of Apparatus

11.1 *Pretest Preparation:*

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11.1.1 Weigh several 200- to 300-g portions of silica gel in airtight containers to the nearest 0.5 g. Record the total weight of the silica gel plus container on each container. Alternatively, the silica gel can be weighed directly in the impinger immediately prior to the train being assembled.

11.1.2 Desiccate the sample filters at $20^{\circ} \pm 5.6^{\circ}\text{C}$ ($68^{\circ} \pm 10^{\circ}\text{F}$) and ambient pressure for 24 to 36 hours, weigh at intervals of at least 6 hours to a constant weight (i.e., $<0.5\text{-mg}$ change from previous weighing), and record results to the nearest 0.1 mg. Alternatively, the filters may be oven-dried at 105°C (220°F) for 2 to 3 hours, desiccated for 2 hours, and weighed.

11.1.3 Clean all sampling train glassware as described in Section 8.10 before each series of tests at a single source. Until the sampling train is assembled for sampling, cover all glassware openings where contamination can occur.

11.2 *Preparation of Sampling Train:*

11.2.1 Assemble the sampling train as shown in Figure 1.

11.2.2 Place 100 mL of the KCl solution (see Section 8.5.1 of this method) in each of the first, second, and third impingers, as indicated in Figure 1.

11.2.3 Place 100 mL of the $\text{HNO}_3\text{-H}_2\text{O}_2$ solution (Section 8.5.2 of this method) in the fourth impinger, as indicated in Figure 1.

11.2.4 Place 100 mL of the $\text{H}_2\text{SO}_4\text{-KMnO}_4$ absorbing solution (see Section 8.5.3 of this method) in each of the fifth, sixth, and seventh impingers, as indicated in Figure 1.

11.2.5 Transfer approximately 200 to 300 g of silica gel from its container to the last impinger, as indicated in Figure 1.

11.2.6 Prior to final train assembly, weigh and record the weight of each impinger. This information is required to calculate the moisture content of the sampled flue gas.

11.2.7 To ensure leak-free sampling train connections and to prevent possible sample contamination problems, use PTFE tape, PTFE-coated O-rings, or other noncontaminating material.

11.2.8 Place a weighed filter in the filter holder using a tweezer or clean disposable surgical gloves.

11.2.9 Install the selected nozzle using a noncontaminating rubber-type O-ring or equivalent when stack temperatures are less than 260°C (500°F) and an alternative gasket

material when temperatures are higher. Other connecting systems, such as PTFE ferrules or ground glass joints, may also be used on the probe and nozzle.

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11.2.10 Mark the probe with heat-resistant tape or by some other method to denote the proper distance into the stack or duct for each sampling point.

11.2.11 Place crushed or cubed ice around the impingers.

11.2.12 *Leak-Check Procedures.* Follow the leak-check procedures given in Section 4.1.4.1 (Pretest Leak Check), Section 4.1.4.2 (Leak Checks During the Sample Run), and Section 4.1.4.3 (Posttest Leak Checks) of EPA Method 5 or 17.

Note 2—If the flue gas temperature at the sampling location is greater than 260°C (above the temperature where PTFE or rubber-type seals can be used), the posttest leak check is determined beginning at the front end of the probe (does not include nozzle or sample filter holder for EPA Method 17).

12. Calibration and Standardization

12.1 *Sampling Train Calibration:*

12.1.1 *Probe Nozzle*—Refer to Sections 2.1.1 of either EPA Method 5 or 17.

12.1.2 *Pitot Tube*—Refer to Section 4 of EPA Method 2.

12.1.3 *Metering System*—Refer to Section 5.3 of either EPA Method 5 or 17.

12.1.4 *Probe Heater*—Refer to Section 7.1.7.1 and 7.1.7.2 of this method.

12.1.5 *Temperature Gauges*—Refer to Section 4.3 of EPA Method 2.

12.1.6 *Leak Check of the Metering System*—Refer to Section 5.6 of EPA Method 5 or Section 5.5 of EPA Method 17.

12.1.7 *Barometer*—Calibrate the barometer to be used against a mercury barometer.

12.2 *Atomic Absorption or Atomic Fluorescence Spectrometer Calibration*—Perform instrument setup and optimization according to the manufacturer's specifications. Cold-vapor generation of mercury is performed via addition of stannous chloride solution to reduce oxidized mercury to its elemental state. The mercury-laden solution is then purged with a carrier gas into the atomic absorption cell. This procedure is used to calibrate the instrument using 10%^{v/v} HCl as the blank along with the standards described in Section 8.9.3. Calibration is verified by analyzing the QC standard prepared according to Section 8.9.4 of this method.

13. Procedures

13.1 *Sampling Train Operation:*

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13.1.1 Maintain an isokinetic sampling rate within 10% of true isokinetic. For an EPA Method 5 configuration, maintain sample filter exit gas stream temperatures and probe within $\pm 15^{\circ}\text{C}$ of the flue gas temperature at the sampling location. However, at no time, regardless of the sample configuration, must the sample filter, probe, or connecting umbilical cord temperature be lower than 120°C .

13.1.2 Record the data, as indicated in Figure 2, at least once at each sample point but not less than once every 5 minutes.

13.1.3 Record the dry gas meter reading at the beginning of a sampling run, the beginning and end of each sampling time increment, before and after each leak check, and when sampling is halted.

13.1.4 Level and zero the manometer. Periodically check the manometer level and zero, because it may drift during the test period.

13.1.5 Clean the port holes prior to the sampling run.

13.1.6 Remove the nozzle cap. Verify that the filter and probe heating systems are up to temperature and that the pitot tube and probe are properly positioned.

Note 3—For an EPA Method 5 configuration, prior to starting the gas flow through the system, the sample filter exit gas temperature may not be at the hot box temperature. However, if the system is set up correctly, once flow is established, the sample filter exit gas temperature will quickly come to equilibrium.

13.1.7 Start the pump. Position the nozzle at the first traverse point with the nozzle tip pointing in the direction of flow. Seal the openings around the probe and port hole to prevent unrepresentative dilution of the gas stream. Read the pitot tube manometer, start the stopwatch, open and adjust the control valve until the isokinetic sampling rate is obtained (refer to Section 4.1.5 from either EPA Method 5 or 17 for information on isokinetic sampling rate computations), and maintain the isokinetic rate at all points throughout the sampling period.

13.1.8 When sampling at one traverse point has been completed, move the probe to the next traverse point as quickly as possible. Close the coarse adjust valve, and shut the pump off when transferring the probe from one sample port to another. Exclude the time required to transfer the probe from one port to another from the total sampling time.

13.1.9 Traverse the stack cross section, as required by EPA Method 1.

13.1.10 During sampling, periodically check and, if necessary, adjust the probe and filter exit sample gas temperatures, as well as the zero of the manometer.

13.1.11 Add more ice, if necessary, to maintain a temperature of $<20^{\circ}\text{C}$ (68°F) at the condenser/silica gel outlet.

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13.1.12 Replace the filter assembly if the pressure drop across the filter becomes such that maintaining isokinetic sampling is no longer possible. Conduct a leak check (refer to EPA Method 5 or 17, Section 4.1.4.2) before installing a new filter assembly. The total particulate weight and determination of particle-bound mercury includes all filter assembly catches.

13.1.13 In the unlikely event depletion of KMnO_4 via reduction reactions with flue gas constituents other than elemental mercury occurs, it may render it impossible to sample for the desired minimum time. This problem is indicated by the complete bleaching of the purple color of the acidified permanganate solution. If the purple color is lost in the first two H_2SO_4 - KMnO_4 impingers, then the sample must be repeated. If the gas stream is known to contain large amounts of reducing constituents (i.e., >2500 ppm SO_2) or breakthrough has occurred in previous sampling runs, then the following modification is suggested: the amount of HNO_3 - H_2O_2 ($10\% \text{V/V}$) in the fourth impinger should be doubled, and/or a second HNO_3 - H_2O_2 impinger should be used to increase the oxidation capacity for reducing gas components prior to the H_2SO_4 - KMnO_4 impingers.

13.1.14 Use a single train for the entire sample run, except when simultaneous sampling is required in two or more separate ducts or at two or more different locations within the same duct or when equipment failure necessitates a change of trains.

13.1.15 At the end of a sample run, turn off the coarse adjust valve, remove the probe and nozzle from the stack, record the final dry gas meter reading, and conduct a posttest leak check, as described in Section 4.1.4.3 of EPA Method 5. Also, leak-check the Pitot lines as described in EPA Method 2, Section 3.1. The lines must pass the leak check to validate the velocity head data.

13.1.16 Calculate percent isokinetic to determine whether the run was valid or another test run should be performed (refer to Section 14.6 of this method).

13.2 Sample Recovery:

13.2.1 Allow the probe to cool before proceeding with sample recovery. When the probe can be safely handled, wipe off all external particulate matter near the tip of the probe nozzle, and place a rinsed, noncontaminating cap over the probe nozzle to prevent losing or gaining particulate matter. Do not cap the probe tip tightly while the sampling train is cooling; a vacuum can form in the filter holder, with the undesired result of drawing liquid from the impingers onto the filter.

13.2.2 Before moving the sampling train to the cleanup site, remove the probe from the sampling train, and cap the open outlet. Be careful not to lose any condensate that may be present. Cap the filter inlet where the probe was fastened. Remove the umbilical cord from the last impinger, and cap the impinger. Cap the filter holder outlet and impinger inlet. Use

noncontaminating caps, such as ground-glass stoppers, plastic caps, serum caps, or PTFE tape, to close these openings.

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13.2.3 Alternatively, the following procedure may be used to disassemble the train before the probe and filter holder/oven are completely cooled. Initially disconnect the filter holder outlet/impinger inlet, and loosely cap the open ends. Then disconnect the probe from the filter holder or cyclone inlet, and loosely cap the open ends. Cap the probe tip, and remove the umbilical cord as previously described.

13.2.4 Transfer the probe and filter-impinger assembly to a clean area that is protected from the wind and other potential causes of contamination or loss of sample. Inspect the train before and during disassembly, and note any abnormal conditions.

13.2.5 The impinger train sample recovery scheme is illustrated in Figure 3.

13.2.6 *Container 1 (Sample Filter)*—Carefully remove the sample filter from the filter holder so as not to lose any ash, weigh filter and ash, and place the filter in a labeled petri dish container. To handle the filter, use either acid-washed polypropylene or PTFE-coated tweezers or clean, disposable surgical gloves rinsed with water and dried. If it is necessary to fold the filter, make certain the particulate cake is inside the fold. Transfer any particulate matter or filter fibers that adhere to the filter holder gasket to the filter in the petri dish. A dry (acid-cleaned) nonmetallic bristle brush should be used to remove any remaining particulate matter. Do not use any metal-containing materials when recovering this train. Immediately cover and seal the labeled petri dish.

13.2.7 *Container 2/2a (All Rinses in Front of the Sample Filter)*

13.2.7.1 *Case 1: Includes Gravimetric Particulate Determination in Addition to Mercury*

Quantitatively recover particulate matter and any condensate from all components prior to the sample filter. A nonmetallic brush may be used for removing particulate matter. All front-half components (all components prior to the sample filter) are then rinsed with acetone as outlined in EPA Method 5 or 17. The acetone rinse is then placed into a container (Container 2a) for which the tare weight has been recorded. Container 2a, with a ribbed watch glass over the top, is placed in a fume hood until the acetone has completely evaporated. After the front-half components have been rinsed with acetone, then rinse these components with 0.1 N HNO₃. The 0.1 N HNO₃ rinse is placed in Container 2.

13.2.7.2 *Case 2: Mercury Determination Only (No Acetone Rinse)*

Quantitatively recover particulate matter and any condensate from all components prior to the sample filter. A nonmetallic brush may be used for removing particulate matter. The front-half components are then rinsed with 0.1 N HNO₃, and this rinse is placed in Container 2.

13.2.8 *Container 3 (Impingers 1 through 3, KCl Impinger Contents and Rinses):*

13.2.8.1 Dry the exterior surfaces of Impingers 1, 2, and 3. Then weigh and record the weight of each impinger (to the nearest 0.5 g).

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13.2.8.2 Clean the filter support, the back half of the filter housing, and connecting glassware by thoroughly rinsing with 0.1 N HNO_3 . Pour the rinse into a glass sample Container 3.

13.2.8.3 Add a 5% w/v KMnO_4 solution to each impinger until a purple color remains.

13.2.8.4 Pour all of the liquid from the three KCl impingers into Container 3.

13.2.8.5 Rinse the impingers and connecting glassware with 10% v/v HNO_3 . Although unlikely, if deposits remain on the impinger surfaces, remove them by doing another 10% v/v HNO_3 rinse that has a very small amount (several drops) of 10% w/v hydroxylamine sulfate solution added to each of the KCl impingers. Add these rinses to Container 3. If the solution in Container 3 becomes clear, add a small amount of the 5% w/v KMnO_4 solution until a pink or slightly purple color is obtained. Check again after 90 min to ensure the purple color remains.

13.2.8.6 Perform a final rinse of the impingers and connecting glassware with 0.1 N HNO_3 , and add to Container 3.

13.2.8.7 Do a final rinse of all glass components with water which is discarded.

13.2.8.8 Mark the height of the fluid level in Container 3, seal, and clearly label the contents.

13.2.9 *Container 4 (Impinger 4, HNO_3 - H_2O_2 Impinger Contents and Rinses):*

13.2.9.1 Dry the exterior surfaces of Impinger 4. Then weigh and record the weight of this impinger (to the nearest 0.5 g).

13.2.9.1 Pour the HNO_3 - H_2O_2 absorbing solution into sample Container 4.

13.2.9.2 Rinse the H_2O_2 - HNO_3 impinger and connecting glassware a minimum of two times with 0.1 N HNO_3 , and pour the rinses into Container 4. Do a final rinse with water and discard water.

13.2.10 *Container 5 (Impingers 5 through 7, H_2SO_4 - KMnO_4 Impinger Contents and Rinses):*

13.2.10.1 Dry the exterior surfaces of Impingers 5, 6, and 7. Then weigh and record the weight of each impinger (to the nearest 0.5 g).

13.2.10.2 Pour all of the liquid from the three H_2SO_4 - KMnO_4 impingers into a glass sample Container 5.

13.2.10.3 Rinse the H_2SO_4 - KMnO_4 impingers and connecting glassware a minimum of two times with 0.1 N HNO_3 , and pour the rinses into Container 5. If deposits remain on the impinger surfaces, after the two rinses, remove them by doing a third rinse with 0.1 N HNO_3 and several drops hydroxylamine sulfate. On a drop by drop basis add more hydroxylamine sulfate until the deposit are removed. Add these rinses to Container 5. If the solution in Container 5 becomes clear, add small amounts of H_2SO_4 - KMnO_4 solution until a pink or slightly purple color is obtained.

13.2.10.4 Perform a final 0.1 N HNO_3 rinse of the impingers and connecting glassware follow by a water rinse. The 0.1 N HNO_3 rinse is added to Container 5, and the water rinse is discarded.

13.2.10.5 Mark the height of the fluid level, seal the container, and clearly label the contents.

Note 4—As stated earlier in the warning in Section 9.1.1, pressure can build up in the sample storage flask because of the potential reaction of KMnO_4 with acid. Do not fill the container completely, and take precautions to relieve excess pressure.

13.2.11 *Container 6 (Impinger 8, Silica Gel Impinger Contents):*

13.2.11.1 Dry the exterior surfaces of Impinger 8. Then weigh and record the weight of this impinger (to the nearest 0.5 g).

13.2.11.2 Note the color of the indicating silica gel to determine whether it has been completely spent, and make a notation of its condition. If spent, the silica gel must be either regenerated or disposed of.

13.2.12 *Solution Blanks (Containers 7–11)*

Solution blanks are taken each time new reagents are prepared.

13.2.12.1 *Container 7 (0.1 N HNO_3 Blank)*—Place 50 mL of the 0.1 N HNO_3 solution used in the sample recovery process into a properly labeled container. Seal the container.

13.2.12.2 *Container 8 (1 N KCl Blank)*—Place 50 mL of the 1 N KCl solution used as the impinger solution into a properly labeled container. Seal the container.

13.2.12.3 *Container 9 (5% HNO_3 –10% H_2O_2 Blank)*—Place 50 mL of the HNO_3 - H_2O_2 solution used as the nitric acid impinger reagent into a properly labeled container. Seal the container.

13.2.12.4 *Container 10 (H_2SO_4 - KMnO_4 Blank)*—Place 50 mL of the H_2SO_4 - KMnO_4 solution used as the impinger solution in the sample recovery process into a properly labeled container. Refer to Note 4 in Section 13.2.10.5 of this method.

13.2.12.5 *Container 11 (10% w/v Hydroxylamine Sulfate Blank)*—Place 100 mL of hydroxylamine sulfate solution into a properly labeled sample container. Seal the container.

13.2.13 *Container 12 (Sample Filter Blank)*—Once during each field test, place into a properly labeled petri dish three unused blank filters from the same lot as the sampling filters. Seal the petri dish.

13.2.14 After all of the samples have been recovered, they must be analyzed within 45 days.

13.2.15 After all impingers and connectors have been properly rinsed and the solutions recovered, the glassware should be cleaned according to the procedures in Section 8.10 or triple-rinsed with 10% v/v HNO₃ followed by a rinsing with water. If a new source is to be sampled or if there are any brown stains on the glassware, then the glassware must be cleaned according to procedures in Section 8.10 of this method. If multiple sites are to be sampled during a single mobilization, an exception to this procedure will be allowed. In this case, a triple rinsing of the glassware with 10% v/v HNO₃ solution followed by a water rinse prior to sampling can be used as an alternative to the procedures in Section 8.10. However, if there are any brown stains on the glassware, then the glassware must be cleaned according to procedures in Section 8.10 of this method.

13.3 *Sample Preparation:*

13.3.1 *Ash Sample (Containers 1 and 2)*

13.3.1.1 *Case 1: Includes Gravimetric Particulate Determination in Addition to Mercury*—The gravimetric particulate loading is determined from the mass of the ash on the filter (Container 1) and the residual particulate from the acetone rinse (Container 2a), as outlined in EPA Method 5 or 17. If a large amount of ash is on the filter, carefully remove the ash to create a raw ash sample from which a representative weighed aliquot can be taken for digestion. If the mass of ash collected on the filter is small (less than 0.5 g), digest the entire filter along with the ash. Dissolve the residual particulate from Container 2a using concentrated HNO₃. This solution is then added to Container 2 (0.1 N HNO₃ probe rinse). The ash material from Container 1 is then digested using the procedures described in Section 13.3.2 of this method. The same procedure is used to determine the mercury on the sample filter blank.

Use a modification of EPA SW 846 7470 to digest the sample in Container 2 prior to analysis. The main modification is that the volumes of reagents and sample have been reduced tenfold to reduce waste. This reduction in reagent volume is acceptable because modern dedicated mercury analyzers do not require the large volumes that previous manual methods required. Transfer a 10-mL aliquot of the sample to a digestion tube with a screw cap.

13.3.1.2 *Case 2: Mercury Determination Only*—The same procedures are followed as described previously in Section 13.3.1.1 with the exception that there is no Container 2a.

13.3.2 *Ash Digestion*—Accomplish the complete dissolution of ash by one of the following methods or an equivalent alternative method. The following methods are for the dissolution of inorganic samples, such as ash or sediments, when an analysis of trace elements including mercury is done.

13.3.2.1 *Microwave Digestion*—The use of this method assumes proper training in microwave digestion techniques. In addition, this method is tailored for a CEM (continuous emission monitor) microwave digestion system. A 0.5-g ash sample, accurately weighed to 0.0001 g, is placed in a PTFE microwave digestion vessel with 3 mL of concentrated HF, 3 mL of concentrated HNO₃, and 3 mL of concentrated HCl. The vessel is sealed and placed in the microwave (along with other vessels). The vessels are slowly heated to a pressure of 347 kPa (50 psi), which is held for 5 minutes, followed by heating to a pressure of 550 kPa (80 psi), which is held for 20 minutes. The vessels are allowed to cool to room temperature before venting. 15 mL of 4%^{w/v} boric acid is added to each vessel. The vessels are sealed and placed in the microwave again. The vessels are slowly heated back to a pressure of 347 kPa (50 psi) and held for 10 minutes. The vessels are again allowed to cool to room temperature before venting. The contents of each vessel are quantitatively transferred to a 50-mL PMP or polypropylene (PP) volumetric flask and diluted; note that care must be taken in adding water to a strong acid solution.

13.3.2.2 *Conventional Digestion*—The use of this method assumes proper training in PTFE bomb digestion techniques. Place a 0.5-g ash sample, accurately weighed to 0.0001 g, in a PTFE digestion vessel with 7 mL of concentrated HF and 5 mL of aqua regia. Seal the vessel, and place it in an oven or water bath at 90°C for a minimum of 8 hours (these may be heated overnight). Cool the vessel to room temperature before venting. Add 3.5 g of boric acid and 40 mL of water to each vessel. Seal the vessels, and place them in the oven or water bath for an additional 1 hour. Cool the vessels again to room temperature before venting. Quantitatively transfer the contents of each vessel to a 100-mL PMP, PP, or glass volumetric flask and dilute. Note that care must be taken in adding water to a strong acid solution.

13.3.3 *Preparation of Aqueous KCl Impinger Solution (Containers 3 and 8)*—Dilute sample in a 500-mL volumetric flask to volume with water, and mix. Use a modification of EPA SW 846 7470 to digest the sample prior to analysis. The main modification is that the volumes of reagents and sample have been reduced tenfold to reduce waste. This reduction in reagent volume is acceptable because modern dedicated mercury analyzers do not require the large volumes that previous manual methods required. Transfer a 10-mL aliquot of the sample to a digestion tube with a screw cap. Add 0.5 mL of concentrated H₂SO₄, 0.25 mL of concentrated HNO₃, and 1.5 mL of 5%^{w/v} KMnO₄ solution. Mix the solution, and allow it to stand for 15 minutes. Add 0.75 mL of 5%^{w/v} K₂S₂O₈ solution, and loosely cap the tube. Place the tube in a dry block heater or water bath equipped with a temperature probe, and heat to 95°C. Do not allow the temperature to exceed 95°C. Hold the sample at 95°C for 2 hours before allowing it to cool to room temperature. The purple color from the added KMnO₄ solution must remain throughout the digestion. Clearing of the solution during the heating indicates the depletion of KMnO₄. Prior to analysis, add 1 mL of 10%^{w/v} of solid hydroxylamine sulfate solution to the sample. The sample solution should remain clear after addition of hydroxylamine sulfate.

13.3.4 *Preparation of $\text{HNO}_3\text{-H}_2\text{O}_2$ Impinger Solution (Containers 4 and 9)*—Dilute sample in a 250-mL volumetric flask to volume with water, and mix. Treat the sample with a modified version of EPA SW 846 7470. Modifications to the method are necessary to properly treat the H_2O_2 -containing impinger solution before the analysis with CVAAS. The modifications include the addition of HCl, the use of an ice bath during the KMnO_4 addition, and the slow addition of the KMnO_4 . Transfer a 5-mL aliquot of the sample to a digestion tube with a screw cap. Add 0.25 mL of concentrated HCl, place the tube in an ice bath, and allow it to cool for 15 minutes. The destruction of H_2O_2 is accomplished by slow addition of saturated KMnO_4 solution in 0.25-mL increments along the inside of the digestion tube. The violence of this reaction requires careful, slow addition of the KMnO_4 for safety reasons and to avoid loss of analyte. Cool the sample for 15 minutes in between each addition, and mix the sample prior to each addition. After the first five additions, increase the increments to 0.5 mL. Carry out the addition of KMnO_4 until the solution remains purple, indicating complete reaction of the H_2O_2 . Record the volume of saturated KMnO_4 solution added to the sample. Add 0.75 mL of 5%^{w/v} $\text{K}_2\text{S}_2\text{O}_8$ solution to the sample, and then cap the tube loosely. Place the tubes in a dry block heater or water bath equipped with a temperature probe, and heat to 95°C. Do not allow the temperature to exceed 95°C. Maintain the sample at 95°C for 2 hours before allowing it to cool to room temperature. Note that the purple color due to KMnO_4 must remain throughout the digestion. Clearing of the solution during the heating indicates the depletion of KMnO_4 . Before doing the analysis, add 1 mL 10%^{w/v} of solid hydroxylamine sulfate solution to the sample. The sample should then become clear.

13.3.5 *Preparation of $\text{H}_2\text{SO}_4\text{-KMnO}_4$ Impinger Solution (Containers 5 and 10)*—Prepare the solution immediately prior to analysis. Dissolve by incrementally adding approximately 500 mg of solid hydroxylamine sulfate into the sample until a clear, colorless solution persists. Add the hydroxylamine slowly because of the violence of this reaction. Dilute the sample in a 500-mL volumetric flask to volume with water, and mix. Transfer a 10-mL aliquot of the sample to a digestion tube with a screw cap. Add 0.75 mL of 5%^{w/v} $\text{K}_2\text{S}_2\text{O}_8$ solution to the sample, and then cap the tube loosely. Place the tube in a dry block heater or water bath equipped with a temperature probe, and heat to 95°C. Do not allow the temperature to exceed 95°C. Hold the sample at 95°C for 2 hours before allowing it to cool to room temperature.

13.3.6 *Simplification of the Digestion*—If an acetone rinse was not used for gravimetric particulate determination or it is very clear, there is insignificant organic material present in the sampled gas stream; then the digestion procedure for the $\text{HNO}_3\text{-H}_2\text{O}_2$ and $\text{H}_2\text{SO}_4\text{-KMnO}_4$ impinger solutions may be simplified by omitting the persulfate digest (the addition of $\text{K}_2\text{S}_2\text{O}_8$ and heating). The persulfate digest is performed for the purpose of oxidizing certain organics. Because this method is specific to coal combustion systems where organic compounds are usually insignificant,⁸ this digest may be omitted because the H_2O_2 is sufficient to oxidize most compounds. The decision to omit this procedure should be made based on the gas stream being

⁸ "A Comprehensive Assessment of Toxic Emissions from Coal-Fired Power Plants: Phase I Results from the U.S. Department of Energy Study," Prepared for the U.S. Department of Energy Federal Energy Technology Center, Contract No. DE-FC21-93MC30097, Energy & Environmental Research Center, University of North Dakota, Grand Forks, ND, 1996.

sampled and/or verification that organics resistant to H_2O_2 oxidation are not present. If unsure whether organics are present or if an acetone rinse has been used, then the total digestion procedure is required.

13.3.7 *0.1 N HNO_3 and 10% $^w/v$ Hydroxylamine Sulfate Rinse Solutions (Containers 7 and 11)*—These solutions can be analyzed directly for mercury without any preparation steps.

13.4 *Sample Analysis*—Analyze all of the prepared solutions by CVAAS or CVAFS following the guidelines specified by the instrument manufacturer.

13.4.1 *QA/QC*—For this method, it is important that both the sampling team and analytical people be very well trained in the procedures. This is a complicated method that requires a high-level of sampling and analytical experience. For the sampling portion of the QA/QC procedure, both solution and field blanks are required. It should be noted that if high-quality reagents are used and care is taken in their preparation and in the train assembly, there should be little, if any, mercury measured in either the solution or field blanks.

As stated in Section 13.2.12 of this method, solution blanks will be taken and analyzed every time a new batch of solution is prepared. If mercury is detected in these solution blanks, the concentration is subtracted from the measured sample results. The maximum amount that can be subtracted is either 10% of the measured result or 10 times the instrument detection limit, whichever is less. If the solution blanks are greater than this general guideline, then the results are not valid.

A field blank is performed by assembling a sample train, transporting it to the sampling location during the sampling period, and recovering it as a regular sample. These data are used to ensure that there is no contamination as a result of the sampling activities. A minimum of one field blank at each sampling location must be completed for each test site. Any mercury detected in the field blanks cannot be subtracted from the results. Whether or not the mercury detected in the field blanks is significant is determined based on the QA/QC procedures established prior to the testing.

The QA/QC for the analytical portion of this method is that every sample, after it has been prepared, is to be analyzed in duplicate with every tenth sample analyzed in triplicate. These results must be within 10% of each other. If this is not the case, then the instrument must be recalibrated and the samples reanalyzed. In addition, after every ten samples, a known spike sample must be analyzed. For the ash samples, a certified reference ash sample (may be purchased from NIST) is to be digested and analyzed at least once during the test program. It is also suggested that the QA/QC procedures developed for a test program include submitting, on occasion, spiked mercury samples to the analytical laboratory by either the prime contractor if different from the laboratory or an independent organization.

14. Flue Gas Calculations

14.1 *Dry Gas Volume*—Calculate the dry gas sample volume, $V_{m(\text{std})}$, at standard conditions using Equation 1.

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$$V_{m(std)} = V_m Y \left(\frac{T_{std}}{T_m} \right) \left[\frac{P_{bar} + \Delta H}{P_{std}} \right] = K_1 V_m Y \frac{P_{bar} + \Delta H}{T_m} \quad [\text{Eq. 1}]$$

where:

- P_{bar} = barometric pressure at the sampling site, kPa (in. Hg)
 P_{std} = standard absolute pressure, 101.3 kPa (29.92 in. Hg)
 T_m = absolute average dry gas meter temperature (refer to Figure 2), K (°R)
 T_{std} = standard absolute temperature, 293 K (528°R)
 V_m = volume of gas sample as measured by dry gas meter, dcm (dscf)
 $V_{m(std)}$ = volume of gas sample measured by the dry gas meter, corrected to standard conditions, dscm (dscf)
 Y = dry gas meter calibration factor
 ΔH = average pressure differential across the orifice meter (refer to Figure 2), kPa (in. Hg)
 K_1 = 2.894 K/kPa (17.64°R/in. Hg)

Note 5—Equation 1 can be used as written unless the leakage rate observed during any of the mandatory leak checks (i.e., leak checks conducted prior to component changes or following the test) exceeds the maximum acceptable leakage rate, L_a , equal to 0.00057 m³/min (0.02 cfm) or 4% of the average sampling rate, whichever is less. If the leakage rate observed during the posttest leak check, L_p , or an individual leakage rate observed during the leak check conducted prior to the “ith” component change ($i = 1, 2, 3, \dots, n$), L_i , exceeds L_a , then Equation 1 must be modified as follows:

(a) **Case I.** No component changes made during sampling run. In this case, replace V_m with the expression:

$$[V_m - (L_p - L_a)\theta]$$

where:

- L_p = leakage rate observed during the posttest leak check, m³/min (cfm)
 L_a = maximum acceptable leakage rate for either a pretest leak check or for a leak check following a component change—equal to 0.00057 m³/min (0.02 cfm) or 4% of the average sampling rate, whichever is less
 θ = total sampling time, min

(b) **Case II.** One or more component changes made during the sampling run. In this case, replace V_m with the expression:

$$\left[V_m - (L_1 - L_a)\theta_1 - \sum_{i=1}^n (L_i - L_a)\theta_i - (L_p - L_a)\theta_p \right]$$

where:

θ_i = sampling time interval, from the beginning of a run until the first component change, min
 and substitute only for those leakage rates (L_i or L_p) that exceed L_a .

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14.2 *Volume of Water Vapor*—Calculate the volume of water vapor of the stack gas using Equation 2.

$$V_{w(std)} = \frac{W_{lc} R T_{std}}{M_w P_{std}} = K_2 W_{lc} \quad [\text{Eq. 2}]$$

where:

- M_w = molecular weight of water, 18.0 g/g-mole (18.0 lb/lb-mole)
- R = ideal gas constant, 0.008314 kPa-m³/K-g-mole (21.85 in. Hg-ft³/°R-lb-mole)
- W_{lc} = total weight of liquid collected in impingers and silica gel (refer to Figure 2), g
- $V_{w(std)}$ = volume of water vapor in the gas sample, corrected to standard conditions, scm (scf)
- K_2 = 0.001336 m³/mL (0.04707 ft³/mL)

14.3 *Volume of Moisture*—Calculate the moisture content, B_{ws} , of the stack gas using Equation 3.

$$B_{ws} = \frac{V_{w(std)}}{V_{m(std)} + V_{w(std)}} \quad [\text{Eq. 3}]$$

where:

- B_{ws} = water vapor in the gas stream, proportion by volume

15. Calculations for Particle-Bound, Oxidized, Elemental, and Total Mercury Concentrations:

15.1 Particle-Bound Mercury

15.1.1 *Case 1: Amount of Ash on the Filter is Greater Than 0.5 g*—Calculate the concentration of mercury in µg/g in the ash sample (Hg_{ash}) using Equation 4:

$$Hg_{ash}, \mu\text{g/g} = (\text{IR})(\text{DF}) \quad [\text{Eq. 4}]$$

where:

- IR = instrument reading, µg/L
- DF = dilution factor = (total digested volume, L)/(mass of ash digested, g)

Calculate the amount of mercury in the probe rinse (Hg_{pr} , Container 2) in µg using Equation 5:

$$Hg_{pr}, \mu g = (IR)(V_1)$$

DRAFT [Eq. 4]

where:

IR = instrument reading, $\mu g/L$

V_1 = total volume of probe rinse sample from which sample aliquot was taken, L

Calculate the amount of mercury on the sample filter blank (Hg_{fb}) in the same way using Equation 6.

$$Hg_{fb}, \mu g = (IR)(V_2) \quad [Eq. 6]$$

where:

IR = instrument reading, $\mu g/L$

V_2 = total volume of sample filter blank digest, L

The total amount of particle-bound mercury (Hg_p) is then determined using Equation 7:

$$Hg \text{ (particle)}, \mu g = (Hg_{ash})(W_{ash}) - Hg_{fb} + Hg_{pr} \quad [Eq. 7]$$

where:

W_{ash} = the total ash weight on filter, g

The concentration of particle-bound mercury ($\mu g/dscm$) in the gas stream is then determined using Equation 8:

$$Hg^p, \mu g/dscm = Hg \text{ (particle)} / V_{m(std)} \quad [Eq. 8]$$

where:

$V_{m(std)}$ = is the total volume of dry gas sampled at standard (normal) conditions, dscm

15.1.2 *Case 2: Amount of Ash on the Filter is Less Than 0.5 g*—The calculation is the same as in Case 1 except the entire sample (ash and filter) is digested; therefore, DF in Equation 4 is defined only by the total digested volume. Equations 5–7 remain the same.

15.2 Oxidized Mercury

15.2.1 *KCl Solution (Impingers 1–3)*—Calculate the concentration of mercury in $\mu g/L$ in the KCl impinger solutions using Equation 9:

$$Hg_{KCl}, \mu g/L = (IR)(DF) \quad [Eq. 9]$$

where:

IR = instrument reading, $\mu g/L$

DF = dilution factor, $\frac{V_D + V(H_2SO_4) + V(HNO_3) + V(KMnO_4) + V(K_2S_2O_8) + V(NH_2OH)}{V_D}$ DRAFT

V_D = total digested volume, 10 mL

$V(H_2SO_4)$ = volume of added concentrated H_2SO_4 , 0.5 mL

$V(HNO_3)$ = volume of added concentrated HNO_3 , 0.5 mL

$V(KMnO_4)$ = volume of added 5%^{w/v} $KMnO_4$, 1.5 mL

$V(K_2S_2O_8)$ = volume of added 5%^{w/v} $K_2S_2O_8$, 0.75 mL

$V(NH_2OH)$ = volume of added 10%^{w/v} hydroxylamine sulfate, 1.0 mL

The amount of mercury in the KCl solution blank is calculated in the same way.

15.2.2 *Total Oxidized Mercury (Hg_O)*—is defined by method as the mercury measured in the KCl sample minus the mercury measured in the KCl solution blanks, as shown in Equation 10:

$$Hg_O, \mu g = (Hg_{KCl})(V_3) - (Hg_{Ob})(V_3) \quad [Eq. 10]$$

where:

Hg_{KCl} = Mercury concentration measured in KCl aliquot, $\mu g/L$

V_3 = Total volume of aqueous KCl from which sample aliquot was taken, L

Hg_{Ob} = Mercury concentration measured in KCl solution blank aliquot, $\mu g/L$

The concentration of Hg^{2+} ($\mu g/dscm$) in the gas stream is then determined using Equation 11:

$$Hg^{2+}, \mu g/dscm = Hg_O/V_{m(std)} \quad [Eq. 11]$$

where:

$V_{m(std)}$ is the total volume of dry gas sampled at standard conditions, dscm

15.3 Elemental Mercury

15.3.1 *HNO_3 - H_2O_2 Solution (Impinger 4)*—Calculate the concentration of mercury in $\mu g/L$ in the HNO_3 - H_2O_2 impinger solution using Equation 12:

$$Hg_{H_2O_2}, \mu g/L = (IR)(DF) \quad [Eq. 12]$$

where:

IR = instrument reading, $\mu g/L$

DF = dilution factor = $\frac{V_D + V(HCl) + V(KMnO_4) + V(K_2S_2O_8) + V(NH_2OH)}{V_D}$

V_D = total digested volume, 5 mL

$V(HCl)$ = volume of added concentrated HCl , 0.25 mL

$V(KMnO_4)$ = volume of added saturated $KMnO_4$, mL (volume need to turn sample to a purple color)

$V(K_2S_2O_8)$ = volume of added 5%^{w/v} $K_2S_2O_8$, 0.75 mL (if used)

$V(NH_2OH)$ = volume of added 10%^{w/v} hydroxylamine sulfate, 1.0 mL

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The amount of mercury in the HNO_3 - H_2O_2 solution blank is calculated in the same way.

15.3.2 H_2SO_4 - $KMnO_4$ Solution (Impingers 5-7)—Calculate the concentration of mercury in $\mu g/L$ in the H_2SO_4 - $KMnO_4$ impinger solutions using Equation 13:

$$\text{Mercury, } \mu g/L = (IR)(DF) \quad [Eq. 13]$$

where:

$$DF = \text{dilution factor} = \frac{V_D + V(K_2S_2O_8)}{V_D}$$

IR = instrument reading, $\mu g/L$

V_D = total digested volume, 5 mL

$V(K_2S_2O_8)$ = volume of added 5%^{w/v} $K_2S_2O_8$, 0.75 mL (if used)

The concentration of mercury in the H_2SO_4 - $KMnO_4$ solution blank is calculated in the same way.

15.3.3 Total Elemental Mercury (Hg_E)—is defined by method as the mercury measured in the H_2SO_4 - $KMnO_4$ impingers plus the mercury in the HNO_3 - H_2O_2 impingers minus the solution blanks as shown in Equation 14:

$$Hg_E, \mu g = (Hg_{H_2O_2})(V_4) - (Hg_{Eb1})(V_4) + (Hg_{KMnO_4})(V_5) - (Hg_{Eb2})(V_5) \quad [Eq. 14]$$

where:

$Hg_{H_2O_2}$ = Mercury concentration measured in HNO_3 - H_2O_2 aliquot, $\mu g/L$

V_4 = Total volume of aqueous HNO_3 - H_2O_2 from which sample aliquot was taken, L

Hg_{Eb1} = Mercury concentration measured in HNO_3 - H_2O_2 solution blank aliquot, $\mu g/L$

Hg_{KMnO_4} = Mercury concentration measured in H_2SO_4 - $KMnO_4$ aliquot, $\mu g/L$

V_5 = Total volume of aqueous H_2SO_4 - $KMnO_4$ from which sample aliquot was taken, L

Hg_{Eb2} = Mercury concentration measured in H_2SO_4 - $KMnO_4$ solution blank aliquot, $\mu g/L$

The concentration of Hg^{2+} ($\mu g/dscm$) in the gas stream is then determined using Equation 15:

$$Hg^0, \mu g/dscm = Hg_E/V_{m(std)} \quad [Eq. 15]$$

where:

$V_{m(std)}$ is the total volume of dry gas sampled at standard conditions, dscm

15.4 Total Mercury—Is defined by the method as the sum of the particulate bound mercury, oxidized mercury, and elemental mercury as shown in Equation 16:

$$\text{Hg}(\text{total}), \mu\text{g/dscm} = \text{Hg}^{\text{p}} + \text{Hg}^{2+} + \text{Hg}^0$$

DRAFT [Eq. 16]

16. Precision and Bias

16.1 Precision

16.1.1 Formal evaluation of the Ontario Hydro method was completed with dynamic spiking of Hg^0 and HgCl_2 into a flue gas stream.⁹ The results are shown in Table 1. The relative standard deviation for gaseous elemental mercury and oxidized mercury was found to be less than 11% for mercury concentrations greater than $3 \mu\text{g/Nm}^3$ and less than 34% for mercury concentrations less than $3 \mu\text{g/Nm}^3$. In all cases, the laboratory bias for these tests based on a calculated correction factor was not statistically significant. These values were within the acceptable range, based on the criteria established in EPA Method 301 (% RSD less than 50%).

16.1.2 The precision of particle-bound, oxidized, and elemental mercury sampling method data is influenced by many factors: flue gas concentration, source, procedural, and equipment variables. Strict adherence to the method is necessary to reduce the effect of these variables. Failure to assure a leak-free system, failure to accurately calibrate all indicated system components, failure to select a proper sampling location, failure to thoroughly clean all glassware, and failure to follow prescribed sample recovery, preparation, and analysis procedures can seriously affect the precision of the results.

16.2 Bias

16.2.1 Gaseous mercury species in flue gases that are capable of interacting with fly ash particles collected in the front half of the sampling train can produce a positive particle-bound mercury bias.

16.2.2 Particle-bound mercury existing in the flue gas may vaporize after collection in the front half of the sampling train because of continued exposure to the flue gas sample stream and reduced pressures during the sampling period. Such vaporization would result in a negative particle-bound mercury bias.

⁹ EPRI. "Evaluation of Flue Gas Mercury Speciation Methods," EPRI TR-108988, Electric Power Research Institute, Palo Alto, CA, Dec. 1997.

Table 1

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Results from Formal EPA Method 301 Evaluation Tests for the Ontario Hydro Method

Ontario Hydro Method**	Total Vapor-Phase Mercury			Oxidized Mercury			Elemental Mercury		
	Mean, $\mu\text{g}/\text{Nm}^3$	Std. Dev.	RSD, %	Mean, $\mu\text{g}/\text{Nm}^3$	Std. Dev.	RSD, %	Mean, $\mu\text{g}/\text{Nm}^3$	Std. Dev.	RSD, %
Baseline	23.35	2.05	8.79	21.24	2.13	10.02	2.11	0.65	30.69
Hg ⁰ Spike (15.0 $\mu\text{g}/\text{Nm}^3$)	38.89	2.00	5.13	23.32	2.08	8.94	15.57	1.09	6.97
HgCl ₂ Spike (19.9 $\mu\text{g}/\text{Nm}^3$)	42.88	2.67	6.23	40.22	2.87	7.14	2.66	0.89	33.31

* For each mean result, there were 12 replicate samples (four quadrants)

** The correction factor in all cases was not statically significant and is not shown.

17. **Keywords**—Air toxics, mercury, sampling, speciation

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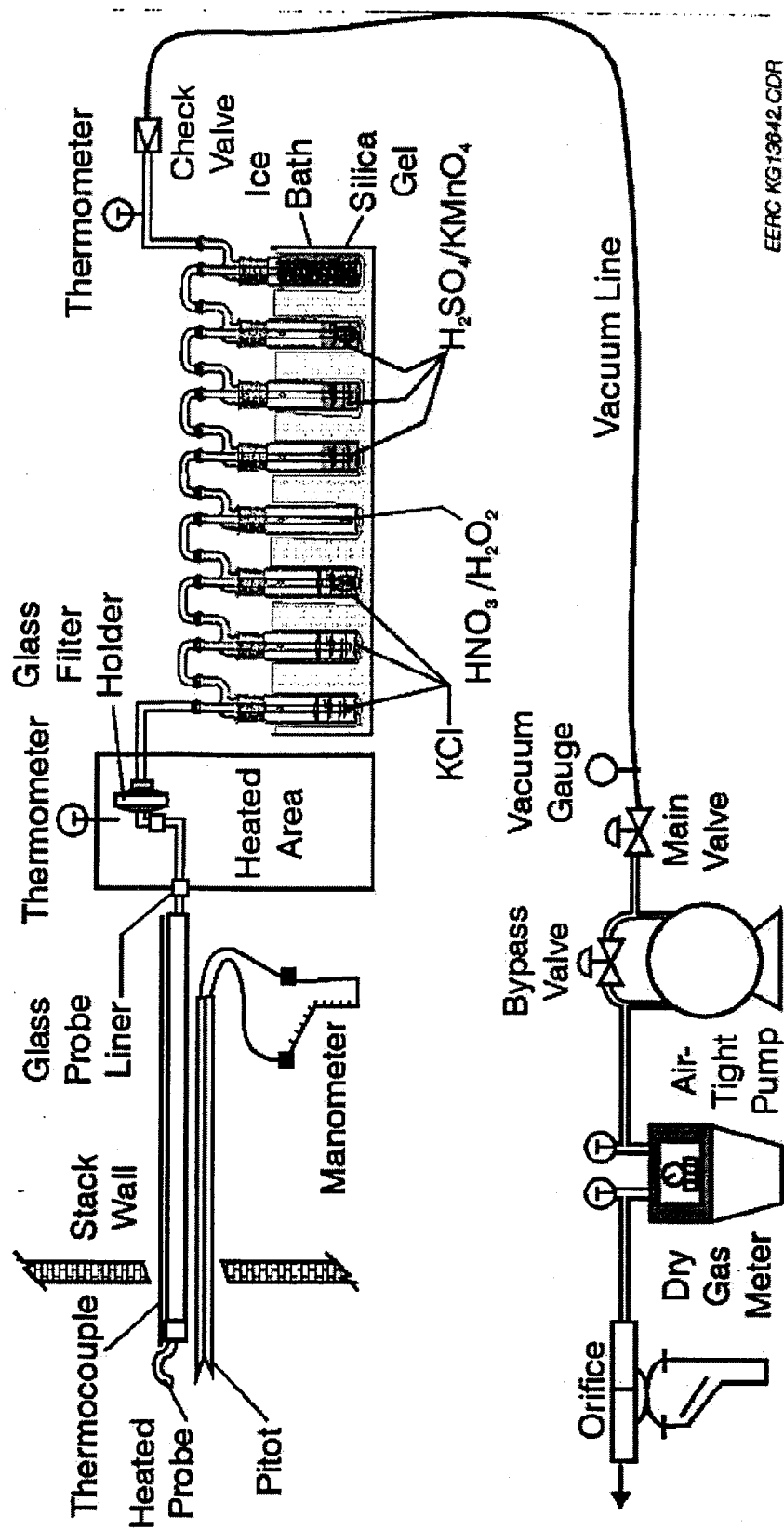



FIG. 1. Schematic of Mercury-Sampling Train in the Method 5 Configuration

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Ambient Temperature °C (°F)
 Barometric Pressure kPa (in. Hg)
 Assumed Moisture, %
 Probe Length, m (ft)
 Nozzle Identification No.
 Average Calibrated Nozzle Diameter, cm (in.)
 Probe Heater Setting °C (°F)
 Leak Rate, m³/min (cfm)
 Static Pressure, kPa (in. Hg)
 Filter No.

Schematic of Stack Cross Section

[illegible]

FIG. 2. Mercury-Sampling Field Data Report

1. Rinse filter holder and connector with 0.1N HNO_3 .
2. Add 5% w/v KMnO_4 to each impinger bottle until purple color remains.
3. Rinse with 10% v/v HNO_3 .
4. Rinse with a very small amount of 10% w/v $\text{NH}_2\text{OH}\cdot\text{H}_2\text{SO}_4$ if brown residue remains.
5. Final rinse with 10% v/v HNO_3 .

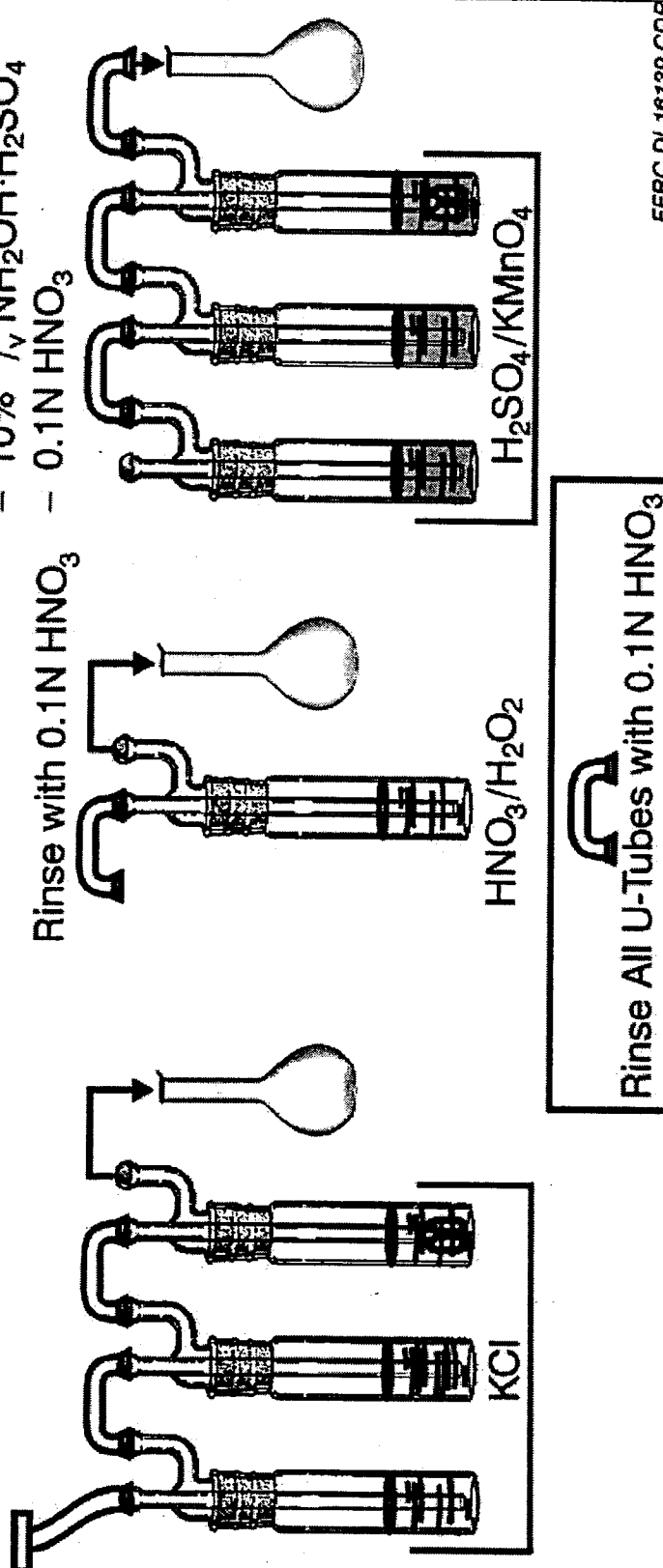


FIG. 3. Sample Recovery Scheme for the Mercury-Sampling Train

BIBLIOGRAPHY OF EPA METHODS REFERENCED

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- (1) Method 303F in *Standard Methods for the Examination of Water and Wastewater*, 15th Edition, 1980. Available from the American Public Health Association, 1015 18th Street N.W., Washington, D.C. 20036.
- (2) EPA Methods 6010, 6020, 7000, 7041, 7060, 7131, 7421, 7470, 7740, and 7841, *Test Methods for Evaluating Solid Waste: Physical/Chemical Methods*. SW-846, Third Edition. September 1988. Office of Solid Waste and Emergency Response, U. S. Environmental Protection Agency, Washington, D.C. 20460.
- (3) EPA Methods 1 through 5, *Code of Federal Regulations*, Title 40, Part 60, Appendix A, July 1, 1991.
- (4) EPA Method 101A, *Code of Federal Regulations*, Title 40, Part 61, Appendix B, July 1, 1991.
- (5) EPA Method 29, *Emission Measurement Technical Information Center*, EMTIC TM-029, April 25, 1996.
- (6) U.S. Environmental Protection Agency "Method 301 – Field Validation of Pollutant Measurement Method from Various Waste Media," In *Code of Federal Regulations*, Title 40, Parts 61 to 80. Washington, DC, USA, Office of the Federal Register, Part 63, Appendix A, pp 324–331, July 1993.

Appendix A.2

Gross Calorific Value of Coal (ASTM D-1989)



Standard Test Method for Gross Calorific Value of Coal and Coke by Microprocessor Controlled Isoperibol Calorimeters¹

This standard is issued under the fixed designation D 1989; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of the gross calorific value of coal and coke by an isoperibol bomb calorimeter using electronic temperature sensors and automatic calorimeter controllers.

1.2 The values stated in SI units are to be regarded as the standard, except as noted otherwise. The values given in parentheses are for information only.

NOTE 1—Conversion to other units is discussed in Appendix X1. Time is expressed in minutes. Mass is expressed in grams.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* For specific hazard statements, see Section 8.

2. Referenced Documents

2.1 ASTM Standards:

- D 121 Terminology of Coal and Coke²
- D 346 Practice for Collection and Preparation of Coke Samples for Laboratory Analysis²
- D 388 Classification of Coals by Rank²
- D 1193 Specification for Reagent Water³
- D 2013 Method of Preparing Coal Samples for Analysis²
- D 3173 Test Method for Moisture in the Analysis Sample of Coal and Coke²
- D 3177 Test Methods for Total Sulfur in the Analysis Sample of Coal and Coke²
- D 3180 Practice for Calculating Coal and Coke Analyses from As-Determined to Different Bases²
- D 4239 Test Methods for Sulfur in the Analysis Sample of Coal and Coke Using High Temperature Tube Furnace Combustion Methods²
- E 144 Practice for Safe Use of Oxygen Combustion Bombs⁴
- E 178 Practice for Dealing with Outlying Observations⁴

3. Terminology

3.1 Definitions:

¹ This test method is under the jurisdiction of ASTM Committee D-5 on Coal and Coke and is the direct responsibility of Subcommittee D05.21 on Methods of Analysis.

Current edition approved July 10, 1997. Published May 1998. Originally published as D 1989 - 91. Last previous edition D 1989 - 96.

² Annual Book of ASTM Standards, Vol 05.05.

³ Annual Book of ASTM Standards, Vol 11.01.

⁴ Annual Book of ASTM Standards, Vol 14.02.

3.1.1 *calorific value, n*—the heat produced by combustion of a unit quantity of a test specimen under specified conditions.

3.1.1.1 *Discussion*—It is expressed in this test method in calories per gram (cal/g), and can also be expressed in British thermal units per pound (Btu/lb), or in the International System of Units (SI) in joules per gram (J/g), when required. The unit equivalents are given in Table 1.

3.1.2 *calorimeter, n*—as used in this test method, not only the bomb and its contents but also includes the bucket, electronic sensing devices, ignition leads, water, and the stirrer when using water.

3.1.3 *calorimeter jacket, n*—the insulating medium surrounding the calorimeter.

3.1.4 *gross calorific value (gross heat of combustion at constant volume), Q_v (gross), n*—see Terminology D 121.

3.1.5 *isoperibol, adj*—a term used in combustion calorimetry meaning constant temperature jacket (environment).

3.1.6 *microprocessor, n*—a central processing chip within the electronic controller section of the apparatus.

3.1.7 *net calorific value (net heat of combustion at constant pressure, Q_p (net), n*—see Terminology D 121.

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *corrected temperature rise*—the temperature change of the calorimeter caused by the process that occurs inside the bomb, that is, the observed temperature change corrected for various affects as noted in 10.4.1.

3.2.2 *energy equivalent, heat capacity, or water equivalent*—the energy required to raise the temperature of the calorimeter one arbitrary unit. This is the quantity that when multiplied by the corrected temperature rise, and then adjusted for extraneous heat effects and divided by the mass of the sample, determines the gross calorific value.

4. Summary of Test Method

4.1 Calorific value is determined in this test method by burning a weighed sample of coal or coke under controlled conditions, in an atmosphere of oxygen, in a calibrated microprocessor controlled isoperibol calorimeter. The calorimeter is calibrated by burning a specified amount of benzoic acid, as defined in 7.3. The calorific value of the test specimen is computed from microprocessor temperature readings made before, during, and after making proper

TABLE 1 Calorific Value

1 Btu = 1055.06 J	1 Btu/lb = 2.326 J/g
1 cal ^A = 4.1868 J	1.8 Btu/lb = 1.0 cal/g

^A International tables calorie.

allowances for heat contributions by other processes. The microprocessor may record these temperatures in either of two modes: a full-temperature method and a temperature extrapolation method.

NOTE 2—Oxidation after sampling and before testing a susceptible low-rank coal or lignite can result in a reduction of calorific value. Unnecessary exposure of the sample to air, or unnecessary delay in analysis from the time of sampling, should be avoided to minimize oxidation.

5. Significance and Use

5.1 When mutually agreed upon between the buyer and seller, the gross calorific value can be used to compute the total calorific content of the shipment of coal, represented by the sample, for commercial purposes.

5.2 The gross calorific value can be used to determine whether the coal meets the regulatory specifications and requirements for industrial fuels.

5.3 The gross calorific value can be used for evaluating the effectiveness of any beneficiation process or for research purposes.

5.4 The gross calorific value is required to classify coals according to procedures specified in Classification D 388.

6. Apparatus and Facilities

6.1 *Test Space*—A room or area free from drafts that can be kept at a reasonably uniform temperature for the time required for the determination. The apparatus shall be shielded from direct sunlight and radiation from other sources. Thermostatic control of room temperature and controlled relative humidity are desirable.

6.2 *Combustion Bomb*, constructed of materials that are not affected sufficiently by the combustion process or products to introduce measurable heat input or to alter the end products. The bomb shall be designed so that all liquid combustion products can be completely recovered by washing the inner surfaces. There shall be no gas leakage. The bomb shall be capable of withstanding a hydrostatic pressure test of 20 MPa (3000 psig) at room temperature without stressing any part beyond its elastic limit.

6.3 *Balance*—A laboratory balance having the capability to accurately weigh the sample to the nearest 0.1 mg. The balance shall be checked periodically to determine its accuracy.

6.4 *Calorimeter Vessel (Bucket)*, made of a suitable metal with a tarnish-resistant coating, with all outer surfaces highly polished. It shall be of such construction that the environment of the calorimeter's entire outer boundaries are maintained at a uniform temperature.

6.5 *Jacket*—A double-walled, air, or water-filled jacket. The calorimeter shall be insulated from the jacket by an air space or an equally satisfactory isolating medium or both. The sides, top, and bottom of the calorimeter vessels shall be positioned approximately 10 mm from the inner wall of the jacket to minimize convection currents. Mechanical supports for the calorimeter vessel shall provide as little thermal conduction as possible. The jacket shall be capable of maintaining the temperature constant to within $\pm 0.1^\circ\text{C}$ of room temperature at a calorimeter temperature 2°C below, and 2°C or more above room temperature throughout the determination. When a water-filled jacket is used, it shall

have a device for stirring the water at a uniform rate.

6.6 *Temperature-Sensing Device*—Thermometers with a precision equal to or better than 0.0001°C . Platinum resistance or other electronic temperature sensors can be used if properly calibrated.

6.7 *Temperature-Measuring Accessories* that measure in degrees Celsius. Equivalent temperatures may be recorded in ohms or other arbitrary units instead of degrees. Consistent units shall be used in standardization as well as in the actual calorific value determination. If arbitrary units other than degrees Celsius are used, the temperature interval shall not vary so as to cause an error greater than 0.001°C .

6.8 *Sample Holder*—An open crucible of platinum, quartz, or base-metal alloy. The base-metal crucibles should be heat-treated for 4 h at 500°C to ensure they are completely oxidized.

6.9 *Ignition Wire*, such as chromium alloy (Chromel C), iron, platinum, or palladium wire that can ignite the sample. It shall be of same length and diameter, or mass, for all calibrations and calorific value determinations.

6.10 *Firing Circuit*, 6- to 24-V ac or dc, required for ignition purposes. A variable transformer connected to an alternating current lighting circuit or batteries can be used.

6.11 *Buret*, having 0.1-mL divisions for the acid titration.

6.12 *Isoperibol Calorimeter, Microprocessor Controlled*—An electronically controlled calorimeter with a central processing unit capable of measuring temperatures, igniting the sample, and calculating the results.

7. Reagents

7.1 *Purity of Reagents*—Reagent grade chemicals conforming to the specification of the Committee on Analytical Reagents of the American Chemical Society shall be used in all tests.⁵

7.2 *Reagent Water*—Reagent water, conforming to Type II of Specification D 1193, shall be used for preparation of reagents and washings of the bomb interior.

7.3 *Benzoic Acid, Standard* ($\text{C}_6\text{H}_5\text{-COOH}$)—Use pellets made from benzoic acid calibrated against the standard material of the National Institute of Standards and Technology.⁶ The value of heat of combustion of benzoic acid, for use in the calibration calculations, shall conform with the certified value.

7.4 *Methyl Orange, Methyl Red, or Methyl Purple Indicator*—Use the indicator to define the titration limits of the acid formed during combustion. The indicator used shall be the same for both calibration and calorific value determinations. Use as a 0.1 % distilled water solution.

7.5 *Oxygen*—Use oxygen manufactured from liquid air only, free of combustible matter, and guaranteed to be greater than 99.5 % pure. Oxygen made by the electrolytic process can contain a small amount of hydrogen and is unfit for use without purification.

⁵ *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

⁶ Benzoic acid to be used as a calibration standard is available from the National Institute of Standards and Technology, Gaithersburg, MD 20899.

7.6 *Sodium Carbonate, Standard Solution* (Na_2CO_3)—Dissolve 3.76 g of sodium carbonate (that has been dried for 24 h at 105°C) in water and dilute to 1 L. One millilitre of this solution is equivalent to 1.0 cal in the acid titration.

7.7 *Wash Water*—Distilled water containing two drops of indicator per 100 mL.

8. Hazards

8.1 In addition to the safety hazards statement given in 1.3, and the equipment manufacturer's installation and operating instructions, special precautions are recommended for safe calorimeter operations by consulting with the calorimeter equipment manufacturer or his certified representative prior to and following the installation. Additional precautions are given in Practice E 144.

8.2 The mass of the coal or coke sample and the pressure of the oxygen admitted to the bomb must not exceed the bomb manufacturer's recommendations.

8.3 Inspect the bomb parts carefully after each use. Check the bomb for thread wear on any closures; if an inspection reveals any wear, replace the worn parts or return the bomb to the factory for testing or replacement of the defective parts. It is good practice to replace the O-rings and seals, inspect screw cap threads, and hydrostatically test the bomb as per the manufacturer's recommendation.

8.4 Equip the oxygen supply cylinder with an approved type of safety device, such as a reducing valve, in addition to the needle valve and pressure gage used in regulating the oxygen to the bomb. Valves, gages, and gaskets must meet industry safety codes. Suitable reducing valves and adapters for 3- to 4-MPa (300- to 500-psi) discharge pressure are obtainable from commercial sources of compressed gas equipment. Check the pressure gage annually for accuracy or after any accidental overpressures that reach maximum gage pressure.

8.5 During ignition of a sample, the operator's body shall not be permitted to be directly exposed to the calorimeter.

8.6 Exercise extreme caution not to exceed the bomb manufacturer's recommendations and avoid damage to the bomb when combustion aids are used. Do not fire loose fluffy material such as unpelleted benzoic acid unless thoroughly mixed with the coal sample.

8.7 Do not fire the bomb if the bomb has been dropped or turned over after loading or if there is evidence of gas leakage when the bomb is submerged in the calorimeter water.

9. Sample

9.1 Pulverize the test specimen to pass a 250- μm (No. 60) sieve prepared in accordance with either Practice D 346 for coke or Method D 2013 for coal.

9.2 Analyze separate test specimens simultaneously for moisture content in accordance with Method D 2013 and Test Method D 3173 so that calculation to other bases can be made.

9.3 Determine sulfur in accordance with Test Methods D 3177 or D 4239.

10. Standardization

10.1 Calibrate the calorimeter (determine the energy equivalent) by combustion of benzoic acid.

10.2 Determine the energy equivalent as the average of a

TABLE 2 Standard Deviations for Calorimeter Standardization—Example^A

Standardization Number	Column A	Column B	Column
	Energy Equivalent, cal/°C	Code to 2449 (Column A-2449)	(Column
1	2450	1	1
2	2448	-1	1
3	2453	4	16
4	2449	0	0
5	2447	-2	4
6	2448	-1	1
7	2446	-3	9
8	2452	3	9
9	2450	1	1
10	2447	-2	4
SUM		0	46

^A Average = $\bar{X} = 2449$ (24490/10).

Variance = $s^2 = \frac{\text{sum of column C} - [(\text{sum column B})^2/n]}{n - 1} = 5.11$.

Standard deviation = $s = \sqrt{\text{variance}} = \sqrt{5.11} = 2.26$.

TABLE 3 Summary of Numerical Requirements^{A,B}

Number of Runs	Maximum Range of Results, cal/°C	Maximum Difference Between X_1 and X_2 , cal/°C
1	...	±3.3
2	4.4	±2.2
4	7.8	±1.7
6	9.4	±1.1
10	11.1	+0.6

^A Test values exceeding table limits require additional runs.

^B Values in this table have been rounded off after statistical calculation and are therefore not precisely in a ratio of 1.8 to 1.0.

X_1 = average of existing standardization and X_2 = average of check runs.

series of ten individual test runs. To be acceptable, the relative standard deviation of the series shall be 0.15 % or less of the average energy equivalent (see Table 2). For this purpose, any individual test may be discarded if there is evidence of incomplete combustion. If, after considering the possibility of outliers using criterion established in Practice E 178, this limit is not met, one should review operation of the calorimeter for any assignable cause which should be corrected before repeating the series.

10.3 Procedure:

10.3.1 Control the mass of the pellets of benzoic acid in each calibration series to obtain the same temperature rise as obtained with typical coal specimens tested in the same laboratory. The usual range of masses is 0.9 to 1.3 g. Weigh the pellet during the same test day and to the nearest 0.0001 g in the sample holder in which it is to be burned and record the mass.

10.3.2 Rinse the bomb with reagent water to lubricate internal seals, dry the exterior surface, and add 1.0 mL of reagent water to the bomb before assembly for a determination.

10.3.3 Connect the measured length, or mass, of ignition wire to the ignition terminals, in accordance with the manufacturer's guidelines (see 6.9.)

10.3.4 Assemble the bomb and charge it with oxygen to a consistent pressure between 2 and 3 MPa (20 and 30 atm). This pressure shall remain the same for each calibration and each calorific value determination. Admit the oxygen slowly into the bomb so as not to displace powdered material from

the sample holder. If the pressure exceeds the specified pressure, detach the filling connection and exhaust the bomb in the usual manner, then discard the sample, as well as the bucket water.

10.3.5 When using a water calorimeter, fill the calorimeter vessel (bucket) with the measured (or weighed) quantity of water adjusted from 1.0 to 2.0°C below the jacket temperature, but not lower than 20°C (Note 4). Use the same mass of water in each test weighed to ± 0.5 g. For 2000-mL calorimeters, the proper quantity can be obtained by use of a volumetric flask calibrated to deliver 2000 ± 0.5 mL. As the density of water varies with temperature, make suitable corrections if the water temperature varies from the temperature at which the flask was calibrated. Place the assembled bomb in the calorimeter vessel. Check that no oxygen bubbles are leaking from the bomb. Place the calorimeter vessel in the jacket; connect the electrodes; place the stirrer, the temperature sensing device, and the cover in position. Start the stirrer and continue to operate it throughout the determination.

NOTE 3—The initial temperature adjustment will ensure a final temperature to be slightly above that of the jacket for 2000-mL calorimeters. Some operators prefer a lower initial temperature so that the final temperature is slightly below that of the jacket. This procedure is also satisfactory. Whichever procedure is used, the same procedure should be used in all tests, including standardization. A small heater may be built into the calorimeter so that the desired starting temperature can be easily attained.

10.3.6 Observations:

10.3.6.1 *Extrapolation Method*—Transfer the bucket, bomb, and calorimeter water to the jacket, finish assembly of the apparatus, and start the calorimeter.

NOTE 4—When jacket water is used, the stirrer should be operating. The calorimeter microprocessor monitors and determines whether the calorimeter temperature drift rate has been constant to within 10^{-4} °C/s for a period of at least 30 s with temperature readings taken at least every 10 s. The microprocessor will fire the charge, record, and correct the temperature rise, using the appropriate heat leak corrections as recommended by the instrument manufacturer. The microprocessor can terminate the test when the observed thermal curve matches the manufacturer's thermal curve which allows extrapolation to a final temperature. The extrapolated temperature rise should have a maximum uncertainty of ± 0.002 °C.

10.3.6.2 *Full-Temperature Development Method*—Transfer the bucket, bomb, and calorimeter water to the jacket, and complete the assembly of the apparatus, and when applicable, start the stirrer. Allow the system to come to equilibrium; then observe the calorimeter temperature at time intervals not exceeding 1 min, or until the rate of change is no greater than ± 0.2 °C. When possible, the calorimeter water temperature should be at the same temperature ± 0.05 °C for every determination at the time of firing. If the U.S. Bureau of Mines method for radiation correction is used (see A1.1.3.3), fire the charge at the start of the sixth time interval; observe and record the temperature t_c and time a . Take the next two readings 0.5 and 1 min after firing. Record subsequent readings at 1-min intervals, or until the temperature differences between successive readings have stabilized within a 0.002°C range over three consecutive 30-s intervals. The time c , and the temperature reading t_c shall be considered as the first readings after the rate of temperature change has become uniform. Estimate resistance thermom-

eter readings to the nearest 0.000 01 Ω.

10.3.7 Open the jacket cover and remove the bomb. Release the pressure at a uniform rate over a period of 1 min. Open the bomb and examine the bomb interior. Discard the test if unburned specimen or sooty deposits are found. Wash the interior of the bomb with wash water containing the titration indicator. Place washings into a beaker and titrate with the standard solution.

10.3.8 Remove and measure, or weigh, the combined pieces of unburned ignition (firing) wire and subtract the remainder from the original length or weight to determine the wire consumed in firing. If the wire is weighed, remove the ball of oxidized metal from the end of each piece of wire before weighing.

10.4 Calculation:

10.4.1 *Temperature Rise*—Using the data obtained as prescribed in 10.3.6, compute the corrected temperature rise t , as follows:

$$t = t_c - t_a + C_s + C_r \quad (1)$$

where:

t = corrected temperature rise, °C;

t_a = initial temperature reading at time of firing, at time a ;

t_c = final temperature reading at time c ;

C_s = thermometer setting correction, if required (see A1.1.2); and

C_r = radiation correction (see A1.1.3).

10.4.2 *Thermochemical Corrections* (see Appendix X2)—Compute the following for each test:

e_1 = correction for the heat of formation of nitric acid (HNO₃), cal.

Each millilitre of standard Na₂CO₃ is equivalent to 1.0 cal; and

e_2 = correction for heat of combustion of firing wire, cal as follows (Note 5):

= 0.23 cal/mm or 1.4 cal/mg for 0.16-mm diameter (No. 34 B&S gage) Chromel C wire.

= 0.27 cal/mm or 1.8 cal/mg for 0.16-mm diameter (No. 34 B&S gage) iron wire.

NOTE 5—There is no correction if platinum or palladium firing wire is used, provided the ignition voltage is constant.

10.4.3 Compute the calorimeter energy equivalent as follows:

$$E = [(g \times H) + e_1 + e_2] \times t^{-1} \quad (2)$$

where:

E = calorimeter energy equivalent, cal/g;

H = heat of combustion of benzoic acid, as stated in the NIST Certificate, cal/g in air;

g = mass (weight in air) of benzoic acid, g;

e_1 = titration correction (10.4.2);

e_2 = fuse wire correction (10.4.2); and

t = corrected temperature rise (10.4.1).

10.5 Repeat the procedure for a total of ten determinations. Compute the standard deviation as illustrated in Table 2. (The percent relative standard deviation is the standard deviation times 100, divided by the average value.)

11. Restandardization

11.1 Make checks on the energy equivalent value (1) after changing the oxygen supply; or (2) after changing any part of the calorimeter; or (3) at least once per month.

11.1.1 If a single new determination differs from the established energy equivalent value by 4 cal/°C (6 Btu/°C),

the new value is suspect, thereby requiring a second test.

11.1.2 The difference between the two new determinations shall not exceed 5 cal/°C (8 Btu/°C) for the energy equivalent, and the average of the two new determinations shall not differ from the established energy equivalent by more than 3 cal/°C (4 Btu/°C) for the energy equivalent. If these requirements are met, do not change the calorimeter standard.

11.1.3 If the requirements given in 11.1.2 are not met, make two more determinations. The range of the four values shall not exceed 8 cal/°C (14 Btu/°C) and the average of the four new determinations shall not differ from the established energy equivalent by more than 2 cal/°C (4 Btu/°C). If these requirements are met, do not change the calorimeter standard.

11.1.4 If the requirements given in 11.1.3 are not met, run a fifth and sixth determination. The range of the six new determinations shall not exceed 10 cal/°C (17 Btu/°C), and the average of the six values shall not differ from the established energy equivalent by more than 2 cal/°C (4 Btu/°C). If these requirements are met, do not change the calorimeter standard.

11.1.5 If the requirements given in 11.1.4 are not met, four more determinations shall be run to complete a series of ten runs. The range of these ten results shall not exceed 12 cal/°C (20 Btu/°C) and the average of the ten new values shall not differ from the established energy equivalent by more than 1 cal/°C (2 Btu/°C). If these requirements are met, do not change the calorimeter standard.

11.1.6 If the requirements of 11.1.5 are not met, evaluate the series of ten runs according to the instructions given in Section 10.

11.2 The summary of the numerical requirements at each stage of restandardization is given in Table 3.

12. Procedure for Coal and Coke Samples (Notes 6 and 7)

12.1 Thoroughly mix the analysis sample of coal or coke in the sample bottle. Weigh out a representative test specimen between 0.5 and 1.0 g to the nearest 0.0001 g. Make each calorific determination in accordance with the procedure described in 10.3.2 through 10.3.8.

NOTE 6—For anthracite, coke, and coal of high ash content that do not burn completely, one of the following procedures is recommended: (1) The mass of the sample can be varied to achieve complete combustion. If the mass is varied, it will be necessary to recalibrate the calorimeter so that the water equivalent will be based on the same temperature rise as that obtained with the new sample mass, and (2) A known amount of benzoic acid can be mixed with the sample. Correction is made for the heat of combustion of benzoic acid when calculating the calorific value of the sample.

NOTE 7—For the calorific value of coke, it is recommended to use 3-MPa (30-atm) pressure for both standardization and analysis.

12.2 Determine the sulfur content of the sample by any of the procedures described in Test Methods D 3177 or D 4239.

13. Calculation

13.1 Compute the corrected temperature rise t , as shown in 10.4.1 where applicable.

13.2 *Thermochemical Correction* (Appendix X2)—Compute the following for each test:

e_1 = correction for the heat of formation of HNO₃ (each millilitre of standard sodium carbonate is equivalent to 1 cal);

e_2 = correction for heat of combustion of ignition wire, cal;
= 0.23 cal/mm or 1.4 cal/mg for 0.16-mm diameter (No. 34 B&S gage) Chromel C wire;
= 0.27 cal/mm or 1.8 cal/mg for 0.16-mm diameter (No. 34 B&S gage) iron wire (Note 5); and
 e_3 = correction for difference between heat of formation of H₂SO₄ from the heat of formation of HNO₃, in calories;
= 13.17 times percent of sulfur in sample times sample mass.

14. Calorific Value (Note 8)

14.1 *Gross Calorific Value*—Calculations can be performed by a microprocessor or one may calculate the gross calorific value (gross heat of combustion at constant volume Q_v (gross) as follows:

$$Q_v \text{ (gross)} = [(t \times E) - e_1 - e_2 - e_3]/g \quad (3)$$

where:

Q_v (gross) = gross calorific value, cal/g;
 t = corrected temperature rise as calculated in 10.4.1, °C;
 E = energy equivalent calculated in 10.4.3, cal/°C;
 e_1, e_2, e_3 = corrections as prescribed in 13.2; and
 g = mass of sample, g.

NOTE 8—This calculation gives calorific value in calories per gram. To obtain calorific value in joules per gram or British thermal units per pound, see Appendix X1.

14.2 *Net Calorific Value*—Calculate the net calorific value (net heat of combustion at a constant pressure), Q_p (net) as follows:

$$Q_p \text{ (net)}_{ar} = Q_v \text{ (gross)}_{ar} - 5.72 (H_{ar} \times 9) \quad (4)$$

where:

$Q_p \text{ (net)}_{ar}$ = net calorific value at constant pressure, cal/g;
 $Q_v \text{ (gross)}_{ar}$ = gross calorific value at constant volume, as-received basis, cal/g; and
 H_{ar} = total hydrogen as-received basis, where hydrogen includes the hydrogen in sample moisture, %.

NOTE 9—Example for converting from the as-determined (air-dried) sample basis to the as-received net calorific value basis:⁷

Calories per gram, as-determined (Cal_{ad})	= 7506
Calories per gram, as-received (Cal_{ar})	= 7056
Moisture, as-determined (M_{ad})	= 2.13
Moisture, as-received (M_{ar})	= 8.00
Hydrogen, as-determined (H_{ad})	= 5.00

To convert H_{ad} to H_{ar} :

$$H_{ar} = \left[(H_{ad} - 0.1119M_{ad}) \times \frac{100 - M_{ar}}{100 - M_{ad}} \right] + 0.1119M_{ar}$$

$$= \left[(5.00 - 0.1119 \times 2.13) \times \frac{100 - 8.00}{100 - 2.13} \right] + 0.1119 \times 8.00$$

$$H_{ar} = 5.37$$

Then:

$$Q_p \text{ (net)}_{ar} = 7056 - 5.72 (5.37 \times 9)$$

$$= 7056 - 276$$

$$= 6780 \text{ cal/g (International Table Calories)}$$

⁷ For a comprehensive theoretical derivation of calculations for converting gross calorific value at constant volume to net calorific value at constant pressure, request Research Report RR:D05-1014.

= 12204 Btu/lb
= 28390 J/g
= 28.39 MJ/kg

Bituminous coals	36 cal/g (64 Btu/lb)
Subbituminous and lignite coals	46 cal/g (83 Btu/lb)
Coke	115 cal/g (206 Btu/lb)

15. Report

15.1 The results of the calorific value can be reported on any of a number of bases, differing from each other in the manner that moisture and ash are treated, and the data must note the reporting base.

15.2 Use the percent moisture in the sample passing a 250- μ m (No. 60) sieve, Test Method D 3173, to calculate the results of the analysis sample to a dry basis.

15.3 Procedures for converting the value obtained on the analysis sample to other bases are described in Practice D 3180.

16. Precision and Bias (Note 8)

16.1 *Precision*—The relative precision of this test method for determination of gross calorific value (Btu) covers the range from 6328 to 8232 cal/g (11 300 to 14 700 Btu/lb) for bituminous coals, 5264 to 7224 cal/g (9400 to 12 900 Btu/lb) for subbituminous and lignite coals, and 7376 cal/g (13 170 Btu/lb) for the average coke value as based upon the analysis of only one coke sample.

16.2 *Repeatability*—The difference in absolute value between two consecutive test results, carried out on the same sample in the same laboratory by the same operator using the same apparatus, should not exceed the repeatability interval (limit) $I(r)$ more than 5 % of such paired values (95 % confidence level). When such a difference is found to exceed the repeatability interval (limit), there is reason to question one or both of the test results. The repeatability limit for this test method on a dry basis is (see Note 10):

Bituminous coals	62 cal/g (110 Btu/lb)
Subbituminous and lignite coals	92 cal/g (164 Btu/lb)
Coke	223 cal/g (399 Btu/lb)

NOTE 10—These limits apply to the relative spread of a measurement that is expressed as a percentage as derived from a statistical evaluation of the round-robin results.

16.4 *Bias*—The equipment used in this test method for measuring gross calorific value has no bias because it is standardized with a compound having a known heat of combustion. This procedure may involve tests that produce varying levels of heat formation not accounted for in standardization. If the thermochemical corrections for heat of formation are not done correctly, a bias may be present in the determination.

17. Keywords

17.1 bomb calorimeter; calorific value; calorimeter; isoperibol bomb calorimeter; microprocessor

ANNEX

(Mandatory Information)

A1. THERMOMETRIC CORRECTIONS

A1.1 Thermometer Corrections:

A1.1.1 It is necessary to make the following individual corrections if not making the corrections would result in an equivalent change of 5.0 Btu or more.

A1.1.2 *Calibration Correction*, shall be made in accordance with the calibration certificate furnished by the calibration authority.

A1.1.3 *Radiation Corrections*—These are used to calculate heat loss to the water jacket. They are based on the Dickinson formula,⁸ the Regnault-Pfaundler formula,⁹ or the U.S. Bureau of Mines method.¹⁰ The same method of determining the radiation correction must be used consistently in calibration and test measurements.

A1.1.3.1 Dickinson Formula:

$$C_r = -r_1(b - i) - r_2(f - b) \quad (A1.1)$$

where:

C_r = radiation correction;

r_1 = rate of rise in temperature per minute in the preliminary period;

r_2 = rate of rise of temperature per minute in the final period (if temperature is falling, r_2 is negative);

t_i = firing temperature;

t_f = final temperature, being the first temperature after which rate of change is constant;

i = time at temperature, t_i , min;

b = time at temperature, $t_i + 0.60(t_f - t_i)$, min; and

f = time at temperature, t_f , min.

A1.1.3.2 Regnault-Pfaundler Formula:

$$C_r = (n \times r_1) + (k \times s) \quad (A1.2)$$

where:

⁸ Dickinson, H. C., *Bulletin*, National Bureau of Standards, Vol 11, 1951, p. 39.

⁹ Pfaundler, L., *Annalen der Physik (Leipzig)*, Vol 129, 1866, p. 102.

¹⁰ "Methods of Analyzing and Testing Coal and Coke," *Bulletin* 638, U.S. Bureau of Mines, 1967, pp. 16-17.

- C_r = radiation correction, °C;
 n = number of minutes in the combustion period;
 s = $(t_n - 1) + 0.05(t_i + t_f) n \times t'$;
 k = $(r_1 - r_2)/(t'' - t')$;
 t' = average temperature during the preliminary period, °C;
 t'' = average temperature during the final period, °C;
 t_1, t_2, t_3 = successive temperature recorded, °C, during the combustion period; and
 $t_n - 1$ = sum of $t_1, t_2, t_3 \dots t_n - 1$.

A1.1.3.3 *Bureau of Mines Method*—A table of radiation corrections can be established so that only the initial and final readings are required to determine the heat value of any fuel. This can be done by performing a series of tests using the procedure described in Section 10, using the following

conditions: Regulate the amount of sample burned so that a series of determinations is made in which different temperature rises are obtained. For all determinations, keep the water jacket temperature constant, fire the bomb at the same initial temperature, and have the same time, $c - a$, elapse (± 2 s) between the initial and final readings. Determine the radiation corrections for each of the series of temperature rises using the Dickinson method (see A1.1.3.1) or the Regnault-Pfaundler method (see A1.1.3.2). These corrections are constant for a given temperature rise. From the series of readings a table or graph is plotted to show radiation correction versus temperature rise. Once the table or graph is established, the radiation corrections can be obtained from it until there is a major change in the equipment.

APPENDIXES

(Nonmandatory Information)

X1. REPORTING RESULTS IN OTHER UNITS

X1.1. Reporting Results in Joules per Gram:

X1.1.1 Because the energy of combustion of the reference material is measured and certified by the National Institute of Standards and Technology in joules per gram, the most straight forward usage of the reference material would lead to the calorific value of the fuel in joules per gram. To carry out this procedure, make changes outlined in X1.1.3 through X1.1.5.

X1.1.2 The gross calorific value can be expressed in joules per gram, calories per gram, or British thermal units per pound. The relationships between these units are given in Table 1.

X1.1.3 For calculating energy equivalent, substitute Eq X1.1 for Eq 2:

$$E' = [(g \times H') + e'_1 + e'_2]t^{-1} \quad (\text{X1.1})$$

where the meanings of the symbols in Eq X1.1 are the same as in Eq 2 except that:

- E' = energy equivalent with units of joules per temperature unit;
 H' = heat of combustion of reference material, with units of joules per gram weight in air (J/g from the certificate for the NIST benzoic acid);
 e'_1, e'_2, e'_3 = corrections with units of joules (see Table X1.1);
 g = mass (weight in air) of benzoic acid, g; and
 t = corrected temperature rise.

X1.1.4 For calculating gross calorific value, substitute Eq

TABLE X1.1 Alternative Thermochemical Correction Factors (Units in Joules)^a

Correction	Multiplication Factor	Multiply By
e'_1 (HNO ₃)	20.0 J/mL	mL of 0.34 N Na ₂ CO ₃
e'_3 (H ₂ SO ₄)	55.2 J/mg	percent of sulfur in sample times mass of sample in grams
e'_2 (fuse wire)	0.95 J/mm	length 0.16-mm diameter of (No. 34 B&S gage) Chromel C wire
or e'_2 (fuse wire)	1.14 J/mm	length 0.16-mm diameter of (No. 34 B&S gage) iron wire
or e'_2 (fuse wire)	6.0 J/mg	mass (milligrams) of Chromel C wire
or e'_2 or (fuse wire)	7.4 J/mg	mass (milligrams) of iron wire

^a To be used in Eqs X1.1 and X1.2 only.

X1.2 for Eq 3:

$$Q_v(\text{gross}) = [(t \times E') - e'_1 - e'_2 - e'_3]g^{-1} \quad (\text{X1.2})$$

where the meanings of the symbols in Eq X1.2 are the same as in Eq 3 except that:

- $Q_v(\text{gross})$ = gross calorific value with units of joules per gram (weight in air);
 E' = energy equivalent with units of joules per temperature unit;
 e'_1, e'_2, e'_3 = corrections with units of joules (see Table X1.1);
 g = mass (weight in air) of benzoic acid, g; and
 t = corrected temperature rise.

X1.1.5 *Precision*—The precision of the procedure in this test method is being determined.

X2. THERMOCHEMICAL CORRECTIONS

X2.1 Energy of Formation of Nitric Acid—A correction, e_1 (10.4.2 and 13.2), is applied for the acid titration. This correction is based on the assumptions (1) that all the acid titrated is HNO_3 formed by the following reaction: $\frac{1}{2} \text{N}_2 (\text{gas}) + \frac{3}{4} \text{O}_2 (\text{gas}) + \frac{1}{2} \text{H}_2\text{O} (\text{liquid}) = \text{HNO}_3$ (in 500-mol H_2O), and (2) that the energy of formation of HNO_3 in approximately 500 mol of water under bomb conditions is -59.0 kJ/mol .¹¹

X2.1.1 A convenient concentration of Na_2CO_3 is 3.76-g $\text{Na}_2\text{CO}_3/1000 \text{ mL}$ which gives $e_1 = V$, where V is the volume of Na_2CO_3 in millilitres. (One millilitre of this solution is equivalent to 1.0 cal in the acid titration.) Use this value (volume equals millilitres) for calculating calorific value in calories per gram. For other units see Table X1.1. When H_2SO_4 is also present, a part of the correction for H_2SO_4 is also present in the e_1 correction and the remainder in the e_3 correction.

X2.2 Energy of Formation of Sulfuric Acid—By definition (see Terminology D 121), the gross calorific value is obtained when the product of the combustion of sulfur in the sample is sulfur dioxide (SO_2) (in grams). However, in actual bomb combustion processes, all the sulfur is found as H_2SO_4 in the bomb washings. A correction e_3 (see 13.2) is applied for the sulfur that is converted to sulfuric acid (H_2SO_4). This correction is based upon the energy of formation of H_2SO_4 in solutions, such as will be present in the bomb at the end of combustion. This energy is taken as -295.0 kJ/mol .¹² A correction of 2 times 59.0 kJ/mol of sulfur was applied in the e_1 correction, so the additional correction necessary is $295.0 - (2 \text{ times } 59.0) = 177 \text{ kJ/mol}$, or 5.52 kJ/g of sulfur in the sample (55.2 J times weight of sample in grams times percent sulfur in sample). This causes e_3 to be 13.17 times

the weight of the sample in grams times percent sulfur in the sample. The factor $23.7 (= 55.2/2.326 \times 1.8)$ for e_3 (see 13.2) is to be used for calculating calorific value in British thermal units per pound. For calculation to other units, see Appendix X1. The values above are based on a coal containing about 5 % sulfur and about 5 % hydrogen. The assumption is also made that the H_2SO_4 is dissolved entirely in the water condensed during combustion of the sample.

X2.2.1 If a 1.0-g sample of such a fuel is burned, the resulting H_2SO_4 condensed with water formed on the walls of the bomb will have a ratio of about 15 mol of water to 1 mol of H_2SO_4 . For this concentration, the energy of the reaction $\text{SO}_2 (\text{gas}) + \frac{1}{2} \text{O}_2 (\text{gas}) + \text{H}_2\text{O} (\text{liquid}) = \text{H}_2\text{SO}_4$ (in 15 mol of H_2O) under the conditions of the bomb process is -295 kJ/mol .¹³ Basing the calculation upon a sample of comparatively large sulfur content reduces the possible overall errors because, for small percents of sulfur, the correction is smaller.

X2.3 Contributions from the burning of the fuse wire shall be in accordance with the directions furnished by the supplier of the wire. For example, the energy of the combustion of No. 34 B&S gage Chromel C wire is 6.0 J/mg or approximately 0.95 J/mm . For calculating e_2 for use in Eqs 2 and 3, these give $e_2 = 0.23$ times length (in millimetres) of wire or 1.4 times weight (in milligrams) of wire. The energy required to melt a platinum wire is constant for each experiment if the same amount of platinum wire or palladium wire is used. As the energy is small, its effect is essentially canceled out in the relationship between the standardization experiments and the calorific value determinations, and this energy can be neglected. The factors listed above for e_2 (10.4.2 and 13.2) are suitable for calculating calorific value in calories per gram. For other units, see Appendix X1.

¹¹ Calculated from data in *NIST Technical Note 270-3*, National Institute of Standards and Technology, Gaithersburg, MD 20899.

¹² Calculated from data in *NIST Circular 500*, National Institute of Standards and Technology, Gaithersburg, MD 20899.

¹³ Mott, R. A., and Parker, C., "Studies in Bomb Calorimetry IX-Formation of Sulfuric Acid," *Fuel*, Vol 37, 1958, p. 371.

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Appendix A.3

Ultimate Analysis of Coal (ASTM D-3176)



Standard Practice for Ultimate Analysis of Coal and Coke¹

This standard is issued under the fixed designation D 3176; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice covers the term ultimate analysis as it is applied to the analysis of coal and coke. The information derived is intended for the general utilization by applicable industries, to provide the basis for evaluation, beneficiation, or for other purposes.

1.2 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

1.3 The values stated in SI units are to be regarded as the standard.

2. Referenced Documents

2.1 ASTM Standards:

D 346 Practice for Collection and Preparation of Coke Samples for Laboratory Analysis²

D 2013 Method of Preparing Coal Samples for Analysis²

D 2234 Practice for Collection of a Gross Sample of Coal²

D 2361 Test Method for Chlorine in Coal²

D 2795 Test Method for Analysis of Coal and Coke Ash²

D 3172 Practice for Proximate Analysis of Coal and Coke²

D 3173 Test Method for Moisture in the Analysis Sample of Coal and Coke²

D 3174 Test Method for Ash in the Analysis Sample of Coal and Coke from Coal²

D 3177 Test Methods for Total Sulfur in the Analysis Sample of Coal and Coke²

D 3178 Test Methods for Carbon and Hydrogen in the Analysis Sample of Coal and Coke²

D 3179 Test Methods for Nitrogen in the Analysis Sample of Coal and Coke²

D 4239 Test Method for Sulfur in the Analysis Sample of Coal and Coke Using High Temperature Tube Furnace Combustion Methods²

3. Terminology

3.1 Definition:

3.1.1 *ultimate analysis*—in the case of coal and coke, the determination of carbon and hydrogen in the material, as found in the gaseous products of its complete combustion, the determination of sulfur, nitrogen, and ash in the material

as a whole, and the calculation of oxygen by difference.

NOTE 1—The determination of phosphorus or chlorine is not by definition a part of the ultimate analysis of coal or coke. See Test Method D 2361 for the determination of chlorine and Test Methods D 2795 for the determination of phosphorus.

NOTE 2—Moisture is not by definition a part of the ultimate analysis of coal or coke but must be determined in order that analytical data may be converted to bases other than that of the analysis sample.

NOTE 3—Inasmuch as some coals contain mineral carbonates, and practically all contain clay or shale containing combined water, a part of the carbon, hydrogen, and oxygen found in the products of combustion may arise from these mineral components.

4. Significance and Use

4.1 Summarizing the ash content and the content of the organic constituents in a specific format under the heading, *Ultimate Analysis*, provides a convenient and uniform system for comparing coals or cokes. This tabulation used with that of *Proximate Analysis* (Method D 3172) permits cursory valuation of coals for use as fuel or in other carbonaceous processes and of cokes for metallurgical purpose.

5. General Requirements

5.1 Coal sample collection shall be in accordance with Test Methods D 2234, and sample preparation shall be in accordance with Method D 2013. Coke sampling and preparation shall be in accordance with Method D 346.

6. Specific Requirements

6.1 *Carbon and Hydrogen*—The carbon and hydrogen determination shall be made in accord with Test Method D 3178.

6.2 *Sulfur*—The sulfur determination shall be made in accordance with Test Methods D 3177 or D 4239.

6.3 *Nitrogen*—The nitrogen determination shall be made in accordance with Test Method D 3179.

6.4 *Ash*—The ash determination shall be made in accordance with Test Method D 3174.

6.5 *Oxygen*—There being no satisfactory direct ASTM test method for determining oxygen, it shall be calculated by subtracting from 100 the sum of the other components of the ultimate analysis. The result so obtained is affected by errors incurred in the other determinations of the ultimate analysis and also by the changes in weight of the ash-forming constituents on ignition. By definition, oxygen calculated as a weight percentage of the analysis sample according to this procedure does not include oxygen in the mineral matter or in the ash, but does include oxygen in the free water (moisture) associated with the analysis sample. See Section 7

¹ This practice is under the jurisdiction of ASTM Committee D-5 on Coal and Coke and is the direct responsibility of Subcommittee D05.21 on Methods of Analysis.

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² *Annual Book of ASTM Standards*, Vol 05.05.

TABLE 1 Procedures for Converting As-Determined Values to Other Bases^A

Reporting Basis Parameter ^B	As-Determined ^C	Dry	As-Received ^{D,E}	
			H_{ar} and Ox_{ar} include H and Ox in Moisture (M_{ar})	H_{ar} and Ox_{ar} do not include H and Ox as M_{ar}
Ash Carbon Nitrogen (P) Sulfur	No corrections (See standard method)	$P_d = P_{ad} \times \left(\frac{100}{100 - M_{ad}} \right)$	$P_{ar} = P_{ad} \times \left(\frac{100 - M_{ar}}{100 - M_{ad}} \right)$	same as column at left
Hydrogen (H)	No corrections (See standard method)	$H_d = (H_{ad} - 0.1119M_{ad})$ $\times \left(\frac{100}{100 - M_{ad}} \right)$	$H_{ar} = \left[(H_{ad} - 0.1119M_{ad}) \right.$ $\times \left(\frac{100 - M_{ar}}{100 - M_{ad}} \right)$ $\left. + 0.1119M_{ar} \right]$	$H_{ar} = (H_{ad} - 0.1119M_{ad})$ $\times \left(\frac{100 - M_{ar}}{100 - M_{ad}} \right)$
Oxygen (Ox)	$Ox_{ad} = 100 - A_{ad} - C_{ad} - H_{ad}$ $- N_{ad} - S_{ad}$	$Ox_d = (Ox_{ad} - 0.8881M_{ad})$ $\times \left(\frac{100}{100 - M_{ad}} \right)$ or $Ox_d = 100 - A_d - C_d - H_d$ $- N_d - S_d$	$Ox_{ar} = \left[(Ox_{ad} - 0.8881M_{ad}) \right.$ $\times \left(\frac{100 - M_{ar}}{100 - M_{ad}} \right)$ $\left. + 0.8881M_{ar} \right]$	$Ox_{ar} = (Ox_{ad} - 0.8881M_{ad})$ $\times \left(\frac{100 - M_{ar}}{100 - M_{ad}} \right)$

^A A = ash, weight %.

M = moisture, weight %.

P = a symbol used interchangeably in the table to refer to ash, or carbon, or nitrogen, or sulfur, weight %.

H = hydrogen, weight %.

Ox = oxygen, weight %.

ad = as-determined from analysis sample.

ar = as received or any other moisture-containing basis (that is, equilibrium capacity moisture basis, as-shipped moisture basis, bed moisture basis) if the appropriate moisture value is substituted for M_{ar} in the formulae, and

d = dry basis.

^B All parameters expressed on a weight percent basis.

^C Hydrogen and oxygen reported on as-determined basis include hydrogen and oxygen in free moisture associated with analysis sample.

^D Alternative procedures are shown, differing on the basis of whether hydrogen and oxygen in the moisture are included or are not included in the report values. A footnote or other means should be employed to indicate the basis used.

^E To convert results to a moisture-containing basis other than as-received, as for example equilibrium capacity moisture, substitute the appropriate moisture value for M_{ar} in the equations.

of this practice for calculating and reporting results on other bases.

6.6 **Moisture**—The moisture determination shall be made in accordance with Test Method D 3173.

7. Calculation and Report

7.1 The results of an ultimate analysis may be reported on any of a number of bases, differing from each other in the manner by which moisture is treated.

7.2 To avoid ambiguity and to provide a means for conversion of data to bases other than the reported basis, it is essential that except for data reported on a dry basis, an appropriate moisture content be given in the data report.

7.3 It is recommended that for data reported on the as-received basis (or any other moist basis) a footnote or some other means be employed in the report to indicate whether the hydrogen and oxygen values reported do include or do not include the hydrogen and oxygen in the free water

(moisture) associated with the sample.

7.4 Procedures for converting ultimate analysis sample data to other bases are presented in Table 1.

7.4.1 Hydrogen and oxygen on the as-determined basis include hydrogen and oxygen in free water (moisture) associated with the analysis sample. However, hydrogen and oxygen values reported on other moisture-containing bases may be reported either as containing or as not containing the hydrogen and oxygen in water (moisture) reported on that basis. Alternative conversion procedures are shown in Table 1.

7.5 An example of ultimate analysis data tabulated for a hypothetical coal on various bases is given in Table 2.

8. Precision

8.1 The permissible differences between two or more determinations shall not exceed the values listed in the precision section of the specific test method for the parameter determined.

TABLE 2 Ultimate Analysis Data

Test Parameter	As-Determined	Dry Basis	As-Received Basis	
	Hydrogen and oxygen include H and O _x in sample moisture (M_{ad})		Hydrogen and oxygen include H and O _x in sample moisture (M_{ar})	Hydrogen and oxygen do not include H and O _x in sample moisture (M_{ar})
Carbon, weight %	60.08	66.02	46.86	46.86
Hydrogen, weight %	5.44	4.87	6.70	3.46
Nitrogen, weight %	0.88	0.97	0.69	0.69
Sulfur, weight %	0.73	0.80	0.57	0.57
Ash, weight %	7.86	8.64	6.13	6.13
Oxygen, weight % (by difference)	25.01	18.70	39.05	13.27
Total %	100.00	100.00	100.00	70.98
Total moisture, weight % (as-received)	...		(29.02)	29.02
Moisture weight % (samples as-determined)	9.00			Total % 100.00

(Air-Dry Loss in accordance with Method D 2013 = 22.00 %)

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This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 100 Barr Harbor Drive, West Conshohocken, PA 19428.

Appendix A.4

Total Mercury in Coal (ASTM D-3684)





Standard Test Method for Total Mercury in Coal by the Oxygen Bomb Combustion/Atomic Absorption Method¹

This standard is issued under the fixed designation D 3684; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method describes a procedure for the analysis of total mercury in coal.

1.2 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* Specific precautionary statements are given in 8.3.1.

1.3 The values stated in SI units (Practice E 380) shall be regarded as the standard.

2. Referenced Documents

2.1 ASTM Standards:

D 1193 Specification for Reagent Water²

D 2013 Method of Preparing Coal Samples for Analysis³

D 3173 Test Method for Moisture in the Analysis Sample of Coal and Coke³

D 3180 Practice for Calculating Coal and Coke Analyses from As-Determined to Different Bases³

D 5142 Test Method for the Proximate Analysis of the Analysis Sample of Coal and Coke by Instrumental Procedures³

E 144 Practice for Safe Use of Oxygen Combustion Bombs⁴

E 380 Practice for the Use of International System of Units (SI) (the Modernized Metric Systems)⁴

3. Summary of Test Method

3.1 Total mercury is determined in this test method by combusting a weighed sample in an oxygen bomb with dilute nitric acid absorbing the mercury vapors. The bomb is rinsed into a reduction vessel with dilute nitric acid, and the mercury is determined by the flameless cold vapor atomic absorption technique.

4. Significance and Use

4.1 The possible emission of mercury that may be found in coal from coal combustion is an environmental concern.

4.2 When test portions are burned according to this procedure, the total mercury is quantitatively retained and is representative of concentrations in the whole coal.

5. Apparatus

5.1 *Combustion Bomb*—The combustion bomb shall be constructed of materials that are not affected by the combustion process or products. The bomb must be designed so that all liquid combustion products can be completely recovered by washing the inner surfaces. There must be no gas leakage during the test. The bomb must be capable of withstanding a hydrostatic pressure test to gage pressure of 20 MPa (approximately 3000 psig) at room temperature without stressing any of the parts beyond the elastic limit.

5.2 *Water Bath*—A container shall be large enough to hold the combustion bomb, and enough cooling water shall be used to dissipate the heat generated during the combustion process. The container should be designed to allow a constant flow of water around the combustion bomb.

5.3 *Combustion Crucibles*—Samples shall be burned in an open crucible of platinum, quartz, or acceptable base-metal alloy.

5.4 *Firing Wire*, 100 mm of either No. 34 B&S (0.160-mm) nickel-chromium alloy, No. 34 B&S iron, or No. 38 B&S (0.101-mm) gage platinum wire.

5.5 *Firing Circuit*—A 6 to 16-V alternating or direct current is required for ignition purposes with an ammeter or pilot light in the circuit to indicate when current is flowing. A step-down transformer connected to an alternating current lighting circuit or batteries may be used. The ignition circuit switch shall be of the momentary double-contact type, normally open, except when held closed by the operator. The switch should be depressed only long enough to fire the charge.

5.6 *Analytical Balance*, with a sensitivity of 0.1 mg.

5.7 *Atomic Absorption Spectrophotometer*, with a flameless cold-vapor mercury analysis system comprised of either a closed recirculating system or an open one-pass system.

5.8 *Reduction Vessels*, Biochemical oxygen demand (BOD) bottles, 300-mL capacity.

6. Reagents

6.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.⁵ Other

¹ This test method is under the jurisdiction of ASTM Committee D-5 on Coal and Coke and is the direct responsibility of Subcommittee D05.29 on Major Elements in Ash and Trace Elements of Coal.

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² *Annual Book of ASTM Standards*, Vol 11.01.

³ *Annual Book of ASTM Standards*, Vol 05.05.

⁴ *Annual Book of ASTM Standards*, Vol 14.02.

⁵ *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

6.2 Reagent Water—Reagent Water, conforming to Type II of Specification D 1193, shall be used for preparation of reagents and washings of the bomb interior.

6.3 Hydroxylamine Hydrochloride Solution (1.5 g/100 mL)—Dissolve 1.5 g of hydroxylamine hydrochloride ($\text{NH}_2\text{OH}\cdot\text{HCl}$) in water and dilute to 100 mL.

6.4 Mercury Standard Stock Solution [1000 ppm (1000 $\mu\text{g/mL}$)]—Dissolve 1.080 g of mercury (II) oxide (HgO) in a minimum volume of HCl (1+1). Dilute to 1 litre with water.

6.5 Mercury Standard Solution [0.1 ppm ($\mu\text{g/mL}$)]—Dilute 0.10 mL of mercury standard stock solution to 1 litre with water. If micropipets are not available, this standard may be prepared by serial dilution of the mercury standard stock solution. Prepare the mercury standard solution daily.

6.6 Nitric Acid (1+9)—Dilute 100 mL of concentrated nitric acid (HNO_3 , sp gr 1.42) to 1 litre with water.

6.7 Oxygen—Oxygen shall be free of combustible matter. Only oxygen manufactured from liquid air, guaranteed to be greater than 99.5 % pure, will meet this requirement.

6.8 Potassium Permanganate Solution (5 g/100 mL)—Dissolve 5 g of potassium permanganate (KMnO_4) in water and dilute to 100 mL.

6.9 Stannous Chloride Solution (10 g/100 mL)—Dissolve 10 g of stannous chloride dihydrate ($\text{SnCl}_2\cdot 2\text{H}_2\text{O}$) in 45 mL of concentrated hydrochloric acid (HCl , sp gr 1.19) and cautiously dilute to 100 mL with water.

7. Sample

7.1 Prepare the analysis sample in accordance with Method D 2013 by pulverizing the material to pass a 250 μm (No. 60) sieve.

7.2 Analyze separate test portions for moisture content in accordance with Test Methods D 3173 or D 5142 so that calculation to other bases can be made.

8. Procedure for Bomb Combustion

8.1 Thoroughly mix the analysis sample of coal in the sample bottle. Weigh a test portion of about 1 g, to the nearest 0.0001 g, into a preignited crucible.

8.2 Transfer 10 mL of HNO_3 (1+9) to the combustion bomb, attach the fuse wire to the bomb electrodes, place the crucible with sample into the electrode support of the bomb, and adjust the fuse wire to contact only the test portion.

8.3 Assemble the bomb in conformance with the manufacturer's directions and charge it with oxygen to a pressure between 2 to 3 MPa (20 and 30 atm). If the oxygen should exceed the specified pressure, stop, detach the filling connection, exhaust the bomb in the usual manner and discard the test portion.

8.3.1 Warning—The following precautions are recommended for safe oxygen bomb operation. Additional precautions are given in Practice E 144.

8.3.1.1 The weight of the test portion and the pressure of the oxygen admitted to the bomb must not exceed the bomb manufacturer's recommendations.

8.3.1.2 Inspect the bomb parts carefully after each use. Check the bomb for thread wear on any closures; if an inspection reveals any wear, replace the worn parts or return

the bomb to the factory for testing or replacement of the defective parts. It is a good practice to replace the o-rings and seals, inspect screw cap threads, and hydrostatically test the bomb as per the manufacturer's recommendations.

8.3.1.3 Equip the oxygen supply cylinder with an approved type of safety device, such as a reducing valve, in addition to the needle valve and pressure gage used in regulating the oxygen feed to the bomb. Valves, gages, and gaskets must meet industry safety code. Suitable reducing valves and adaptors for 2.0 to 3.4-MPa (300 to 500 psi) discharge pressures are obtainable from commercial sources of compressed gas equipment. Check the pressure gage periodically for accuracy.

8.3.1.4 During ignition of a test portion, the operator must not permit any portion of his body to extend over the oxygen bomb.

8.3.1.5 Exercise extreme caution when combustion aids are employed so as not to exceed the bomb manufacturer's recommendations and to avoid damage to the bomb. Do not fire loose fluffy material such as unpeletted benzoic acid, unless thoroughly mixed with the test portion.

8.3.1.6 Admit oxygen slowly into the bomb so as not to blow powdered material from the crucible.

8.3.1.7 Do not fire the bomb if it has been filled to greater than 3 MPa (30 atm) pressure with oxygen, or the bomb has been dropped or turned over after loading, or there is evidence of a gas leak when the bomb is submerged in the oxygen bomb water.

8.4 Place the bomb in the cooling water bath, with water flowing, attaching ignition wires from firing circuits, and ignite the test portion (Warning, 8.3.1). Allow the bomb to remain in the cooling water bath for 10 min to allow temperature equilibration and absorption of soluble vapors.

8.5 Remove the bomb and release the pressure at a uniform rate, such that the operation will require not less than 2 min. Examine the bomb interior and discard the test results if unburned or sooty deposits are found.

8.6 Quantitatively rinse the bomb, electrodes, and crucible into the reduction vessel with several small portions of water. Dilute the contents of the reduction vessel with HNO_3 (1+9) to a total volume of 100 mL. Add KMnO_4 solution dropwise until the permanganate color persists for 60 s.

9. Procedure for Atomic Absorption Analysis

9.1 Align the optical cell in the beam path of the atomic absorption spectrophotometer and optimize the instrument using normal operating conditions as set forth by the instrument manufacturer.

9.2 Prepare standards of 0.10, 0.25, and 0.50 μg of mercury by diluting aliquots of the mercury standard solution to 100 ± 10 mL with HNO_3 (1+9) solution.

9.3 Add KMnO_4 solution dropwise to the standards until the permanganate color persists for 60 s.

9.4 Add 5 mL of hydroxylamine hydrochloride ($\text{NH}_2\text{OH}\cdot\text{HCl}$) solution. When the pink color fades, wait 30 s and add 5 mL of stannous chloride (SnCl_2) solution and immediately connect the reduction flask to the flameless mercury system and determine the absorbance.

9.5 Repeat this procedure (9.4) for unknown test portion solutions (8.6).

9.6 A reagent blank shall be prepared according to 9.2,

9.3, and 9.4, but omit the mercury standard solution in 9.2.

9.7 The absorbance signal is recorded by either a strip chart recorder or read directly from the instrument. An expanded scale can be used to increase the sensitivity.

10. Calculation

10.1 Calculate the concentration of mercury in ppm ($\mu\text{g/g}$) in the analysis sample as follows:

$$\text{Mercury, ppm } (\mu\text{g/g}) = \frac{\left(\frac{C}{A-B}\right)(A_1 - B)}{D} \quad (1)$$

where:

A = signal of standard sample nearest A_1 ,

A_1 = signal of analysis sample,

B = signal of blank sample,

C = total concentration of standard, μg , and

D = sample weight, g.

A standard curve may also be constructed by plotting peak height versus micrograms of mercury and the calculations performed as follows:

$$\text{Mercury, ppm } (\mu\text{g/g}) = W/S \quad (2)$$

where:

W = mercury in sample determined from calibration curve, μg , and

S = sample weight, g.

11. Report

11.1 The results of the mercury analysis may be reported on any number of bases, differing from each other in the manner in which moisture is treated, and the data must note the reporting base.

11.2 Use the percent moisture, as determined by Test Method D 3173 or Test Method D 5142, in the analysis sample passing a 250 μm (No. 60) sieve to calculate the results of the analysis to a dry basis.

11.3 Procedures for converting the value obtained on the

analysis sample to other basis are described in Practice D 3180.

12. Sensitivity

12.1 The detection limit of the test method described above is 0.01 μg assuming a 100 ± 10 mL volume in the reduction vessel.

13. Precision and Bias

13.1 The relative precision of this test method was calculated from test results obtained on coals with a range of 0.05 to 0.2 ppm ($\mu\text{g/g}$) mercury.

13.2 *Repeatability*—Results of two consecutive determinations carried out in the same laboratory by the same operator using the same apparatus should not differ by more than 0.019 ppm ($\mu\text{g/g}$) (Note 1).

13.3 *Reproducibility*—The means of results of duplicate determinations carried out by different laboratories on representative samples taken from the bulk sample after the last stage of reduction should not differ by more than 0.031 ppm ($\mu\text{g/g}$) (Note 1).

13.4 *Bias*—Results obtained with this test method on National Institute of Standards and Technology (NIST) standard reference material 1632 are shown below:

NIST— 0.12 ± 0.02 ppm ($\mu\text{g/g}$)

D 3684— 0.095 ± 0.007 ppm ($\mu\text{g/g}$) (Note 1)

NOTE 1—Values from Test Method D 3684 represent the mean of the means from four separate laboratories,⁶ each of which made four replicate analyses on four separate samples of the coal standard reference material.

14. Keywords

14.1 coal; flameless cold vapor atomic absorption; mercury; oxygen bomb

⁶ Supporting data are available from ASTM Headquarters. Request RR:D05-1002.

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Appendix A.5

Total Chlorine in Coal (ASTM D-4208)



Standard Test Method for Total Chlorine in Coal by the Oxygen Bomb Combustion/Ion Selective Electrode Method¹

This standard is issued under the fixed designation D 4208; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the analysis of total chlorine in coal.

1.2 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

D 1193 Specification for Reagent Water²

D 3173 Test Method for Moisture in the Analysis Sample of Coal and Coke³

D 3180 Practice for Calculating Coal and Coke Analysis from As-Determined to Different Bases³

E 144 Practice for Safe Use of Oxygen Combustion Bombs⁴

3. Summary of Test Method

3.1 Total chlorine is determined in this method by combusting a weighed sample in an oxygen bomb with dilute base adsorbing the chlorine vapors. The bomb is rinsed into a beaker with water and following the addition of an ionic strength adjuster, the chloride is determined by ion-selective electrode.

4. Significance and Use

4.1 The purpose of this test method is to measure the total chlorine content of coal. The chlorine content of coals may be useful in the evaluation of slagging problems, corrosion in engineering processes, and in the total analysis of coal and coke. When coal samples are combusted in accordance with this method, the chlorine is quantitatively retained and is representative of the total chlorine content of the whole coal.

5. Apparatus

5.1 *Combustion Bomb*, constructed of materials that are not affected by the combustion process or products. The bomb must be designed so that all liquid combustion products can be quantitatively recovered by washing the

inner surfaces. There must be no gas leakage during the test. The bomb must be capable of withstanding a hydrostatic-pressure test to 3000 psig (approximately 20 MPa) at room temperature without stressing any part beyond its elastic limit.

5.2 *Water Bath*—A container large enough to hold the combustion bomb and enough cooling water to dissipate the heat generated during the combustion process. The container shall be designed to allow a constant flow of water around the combustion bomb.

5.3 *Combustion Crucibles*—Samples shall be burned in an open crucible of platinum, quartz, or acceptable base-metal alloy.

5.4 *Firing Wire*, 100-mm, nickel-chromium alloy, No. 34B &S gage, or platinum, No. 34 or No. 38B &S gage.

5.5 *Firing Circuit*—A 6 to 16-V alternating or direct current is required for ignition purposes with an ammeter or pilot light in the circuit to indicate when current is flowing. A step-down transformer connected to an alternating-current lighting circuit or batteries can be used. **Caution**—The ignition circuit switch shall be of the momentary double-contact type, normally open, except when held closed by the operator. The switch should be depressed only long enough to fire the charge.

5.6 *Balance*, analytical, with a sensitivity of 0.1 mg.

5.7 *Specific-Ion Meter*—A pH meter with an expandable millivolt scale, specific-ion meter, sensitive to 0.1 mV, suitable for method of standard addition determinations.⁵

5.8 *Electrodes*, chloride-sensing, with the appropriate reference-type electrode as recommended by the manufacturer.

6. Reagents

6.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.⁶ Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

¹ This test method is under the jurisdiction of ASTM Committee D-5 on Coal and Coke and is the direct responsibility of Subcommittee D05.21 on Methods of Analysis.

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² *Annual Book of ASTM Standards*, Vol 11.01.

³ *Annual Book of ASTM Standards*, Vol 05.05.

⁴ *Annual Book of ASTM Standards*, Vol 14.02.

⁵ Midgley, D., and Torrance, K., *Potentiometric Water Analysis*, John Wiley and Sons, 1978.

⁶ *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Anal. Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

6.2 *Purity of Water*, deionized, high-purity, low-specific conductivity, Type II reagent water as defined in Specification D 1193.

6.3 *Ionic Strength Adjuster Solution* (5M NaNO₃)—Dissolve 42.5 g of sodium nitrate in 100 mL water.

6.4 *Sodium Carbonate Solution* (Na₂CO₃) (2 %)—Dissolve 2.0 g of sodium carbonate in 100 mL water.

6.5 *Chloride, Standard Stock Solution* (1000 µg/mL)—Dissolve 1.6486 g of sodium chloride (NaCl) in water and dilute to 1 L. The NaCl should be dried for 1 h at 105°C and cooled to room temperature in a desiccator before weighing.

6.6 *Chloride, Standard Stock Solution* (100 µg/mL)—Dilute 10.0 mL of chloride stock solution to 100 mL in a volumetric flask with water.

6.7 *Oxygen*, free of combustible matter and guaranteed to be 99.99 % pure.

7. Sample

7.1 A convenient sample is the air-dried coal that must be pulverized to pass a No. 60 (250-µm) sieve.

7.2 A separate portion of the analysis sample shall be analyzed simultaneously for moisture content in accordance with Test Method D 3173 if calculation to other than the as-determined basis is desired.

8. Procedure for Bomb Combustion

8.1 Thoroughly mix the analysis sample of coal. Carefully weigh approximately $1\text{g} \pm 0.1\text{mg}$ into a previously ignited crucible in which it is to be combusted.

NOTE 1—For samples in excess of 5 % sulfur, the weight of coal must be reduced to $0.5 \pm 0.1\text{g}$ to ensure that all the acidic vapors produced in the combustion process are quantitatively retained in solution.

8.2 Transfer 5 mL of 2 % Na₂CO₃ solution into the combustion bomb. Attach the fuse wire to the bomb electrodes. Place the crucible with the sample into the electrode support of the bomb, and insert the fuse wire so that it just touches the surface of the sample.

8.3 Assemble the bomb in the normal manner and charge it with oxygen to a pressure between 20 and 30 atm (2 to 3 MPa). If the oxygen should exceed the specified pressure, do not proceed with the combustion. In this case, detach the filling connection, exhaust the bomb in the usual manner, and discard the sample.

NOTE 2—Caution: The following precautions are recommended for safe operations in the use of the oxygen combustion bomb. Additional precautions are given in Recommended Practice E 144, for use of oxygen combustion bombs.

8.3.1 The weight of coal sample and the pressure of the oxygen admitted to the bomb must not exceed the bomb manufacturer's recommendation.

8.3.2 Inspect the bomb parts carefully after each use. Frequently check the threads on the main closure for wear. Replace the cracked or significantly worn parts. Return the bomb to the manufacturer occasionally for inspection and possibly proof testing.

8.3.3 The oxygen supply cylinder should be equipped with an approved type of safety device, such as a reducing valve, in addition to the needle valve and pressure gage used in regulating the oxygen feed to the bomb. Valves, gages, and gaskets must meet industry safety code. Suitable reducing

valves and adaptors for 300 to 500-psi (approximately 3 to 5-MPa) discharge pressure are obtainable from commercial sources of compressed-gas equipment. Check the pressure gage periodically for accuracy.

8.3.4 During ignition of a sample, the operator must not permit any portion of his body to extend over the combustion bomb or its container.

8.3.5 Exercise extreme caution when combustion aids are employed so as not to exceed the bomb manufacturer's recommendations and to avoid damage to the bomb.

8.3.6 Admit oxygen slowly into the bomb to avoid blowing powdered material from the crucible.

8.3.7 Do not fire the bomb if it has been filled to greater than 30 atm (3 MPa) pressure with oxygen, if the bomb has been dropped or turned over after loading, or if there is evidence of a gas leak when the bomb is submerged in the water bath.

8.4 Place the bomb in a cooling water bath, with water moving. Attach the ignition wires from the firing circuits, and ignite the sample. Allow the bomb to remain in the cooling water for 15 min to allow cooling and absorption of soluble vapors within the bomb.

8.5 Remove the bomb and release the pressure at a uniform rate, such that the operation will require not less than 2 min. Examine the bomb interior and discard the test if unburned or sooty deposits are found.

8.6 Thoroughly rinse the bomb, electrodes, and crucible into a 100-mL graduated cylinder with several small washings of water, keeping the volume below 90 mL.

9. Procedure for Ion-Selective Electrode Analysis

9.1 Add 2 mL of the ionic-strength adjustor and adjust the volume to 100 mL with water and transfer to a 250-mL beaker.

NOTE 3—For maximum electrode response, all solutions should be measured at ambient temperatures. Electrode response may also be affected if the membrane is dirty or etched. It is recommended that the electrode membrane be repolished before each use.

9.2 Determine the potential of the solution with a chlorine ion-selective electrode. Add 10.0 mL of the chloride standard solution to the beaker with constant stirring and again determine the potential.

10. Calculation

10.1 Determine the chlorine content of the solution from the change in potential (ΔE) resulting from the addition of the (chloride) standard solution. Calculate the concentration of chlorine in ppm (µg/g) in the analysis sample as follows:

Chlorine, ppm in solution

$$= \frac{V_a C_a}{V_s \left[\left(\text{antilog} \left[\frac{\Delta E}{S} \right] \right) \left(\frac{V_a}{V_s} + 1 \right) - 1 \right]} - C_B \quad (1)$$

$$\text{Chlorine, ppm in sample} = \frac{(\text{chlorine in solution}) V_s}{W_s} \quad (2)$$

where:

V_a = volume of added standard, mL,

C_a = standard concentration, µg/g,

C_B = blank concentration, µg/g,

W_s = weight of sample, g

V_s = volume of sample, mL,
 ΔE = potential change, mV, and
 S = electrode slope constant.

NOTE 4—Microprocessor pH/mV meters (ion meters) perform the necessary calculations and display the ion concentration directly.

NOTE 5—Determine a reagent blank concurrently with the test determination using the same amounts of all reagents and following all steps of the procedure.

NOTE 6—The electrode slope constant may be determined as follows:

(1) Add by pipette, 100 mL of standard solution of concentration C_1 to a 250-mL beaker.

(2) Add 2 mL of the ionic strength adjustor.

(3) Stir the solution and when the electrodes give a steady reading, note the reading, E_1 .

(4) Repeat step 2 with a second solution of concentration, C_2 . Preferably $C_2 = 10 C_1$ and should not be less than $2 C_1$.

(5) Repeat steps 2 and 3, noting the steady reading, E_2 .

(6) Calculate the slope constant S , which should be about -58 mV per tenfold increase in concentration at 20°C .

$$S = \frac{E_1 - E_2}{\log C_1 - \log C_2} \quad (3)$$

11. Report

11.1 The results of the chlorine analysis may be reported on any of a number of basis, differing from each other in the manner by which moisture is treated.

11.2 Use the percent moisture, in accordance with Test Method D 3173, in the analysis sample passing a No. 60 (250- μm) sieve (see 7.2), to calculate the results of the analysis to a dry basis.

11.3 Procedures for converting the value obtained on the analysis sample to other bases are described in Method D 3180.

12. Precision and Bias

12.1 *Precision*—The relative precision of this test method for the determination of chlorine covers the concentration range from 220 to 2100 $\mu\text{g/g}$.

12.1.1 *Repeatability*—The difference in absolute value between two consecutive tests results, carried out on the same sample in the same laboratory by the same operator using the same apparatus, should not exceed the repeatability

interval $I(r)$ more than 5 % of such paired values (95 % confidence level). When such a difference is found to exceed the repeatability interval, there is reason to question one or both of the test results. The repeatability interval may be determined by use of the following equation:

$$I(r) = 48.4 + 0.13 x$$

where x is the average of the two test results.

NOTE 7—This equation applies to the relative spread of a measurement that is expressed as a percentage and is derived from the statistical evaluation of the round-robin results.⁷ *Example:* Duplicate analysis for chlorine gave values of 1014 $\mu\text{g/g}$ and 1046 $\mu\text{g/g}$. The average chlorine value from the duplicate analysis is 1030 $\mu\text{g/g}$ and the calculated repeatability interval $I(r)$ is 85 $\mu\text{g/g}$. The difference between the two values is 32 $\mu\text{g/g}$ and does not exceed the $I(r)$ of 85, therefore, these two values are acceptable at the 95 % confidence level.

12.1.2 *Reproducibility*—The difference in absolute value of replicate determinations, carried out in different laboratories on representative samples prepared from the same bulk sample after the last stage of reduction, should not exceed the reproducibility interval $I(R)$ more than 5 % of such paired values (95 % confidence level). When such a difference is found to exceed the reproducibility interval, there is reason to question one or both of the test results. The reproducibility interval may be determined by use of the following equation.:

$$I(R) = 200 + 0.23 x$$

where x is the average of the two results (see Note 7). *Example:* Duplicate analysis for chlorine in one laboratory gave an average value of 1083 $\mu\text{g/g}$ and a value of 1280 $\mu\text{g/g}$ was obtained in a different laboratory. The between laboratory average chlorine value is 1181 $\mu\text{g/g}$, the calculated $I(R)$ interval is 472 $\mu\text{g/g}$, and the difference between the different laboratory value is 197 $\mu\text{g/g}$. Since this is less than the $I(R)$, these two values are acceptable at the 95 % confidence level.

12.2 *Bias*—Since there is no accepted reference material suitable for determining bias for this test method, no statement of bias is being made.

⁷ Supporting data are available from ASTM Headquarters. Request RR:D05-1005.

The American Society for Testing and Materials takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 100 Barr Harbor Drive, West Conshohocken, PA 19428.

Probe Assembly A-9D (Post-test)
and A-10B only

STACK TEMPERATURE SENSOR CALIBRATION DATA FORM

CLIENT: Avogadro

THERMOCOUPLE NO.: TYPE-K

DATE: 10/25/99

AMBIENT TEMP., °F: 69°

OPERATOR: PG

BAROMETRIC PRESS. (in. Hg): 29.74

CALIBRATOR: EM

REF. (MERCURY-IN-GLASS): 67°

NAME: E. Mirabella / P. Gates

SERIAL #: Ref. RT #'s 1, 2, 3

REFERENCE POINT NUMBER*	SOURCE ^b (SPECIFY)	REFERENCE THERMOMETER TEMPERATURE, °F	THERMOCOUPLE POTENTIOMETER TEMPERATURE, °F	TEMPERATURE DIFFERENCE, ° %
COLD	ICE WATER T-U2 T-U4	10°C = 33.8°F	33°F 35°F	0.16 ✓ 0.24 ✓
MEDIUM	BOILING WATER	-	-	-
HOT	HOT OIL T-9Ds T-9Dp T-10Bs T-10Bp	146°C = 294.8°F	298°F 301°F 300°F 303°F	0.42 ✓ 0.82 ✓ 0.69 ✓ 1.09 ✓
Medium	Oven Temp. T-04 T-06	143°C = 289.4°F 140°C = 284°F	293°F 280°F	0.48 ✓ 0.54 ✓

*EVERY 100°F FOR EACH REFERENCE POINT.

^bTYPE OF CALIBRATION SYSTEM USED.

$$^{\circ} \left[\frac{(\text{REF. TEMP., } ^{\circ}\text{F} + 460) - (\text{TEST THERMOM. TEMP., } ^{\circ}\text{F} + 460)}{\text{REF. TEMP., } ^{\circ}\text{F} + 460} \right] 100 \leq 1.5\%$$

Average Test Temp. = 295°F



Appendix B.3

Quality Assurance and Control Forms

Quality Assurance Report Memorandum

Project 99057, Air Products Stockton Cogen Mercury Speciation

Kevin J. Crosby

January 19, 2000

I was assigned as Quality Assurance Officer to the Mercury Speciation project. My responsibilities included observation of the field operations for sampling and sample recovery, review of the calibration data for the apparatus, and review and verification of the test results. Most of these tasks are documented on the QA forms that are included with this memorandum as an Appendix to the report. Other QA tasks are described here.

The tests were conducted according to the Test Protocol, Revision 1, submitted on September 30, 1999. The only changes noted are listed here:

- Pitot tubes calibrated by dimensional measurement, rather than by wind tunnel.
- A full traverse for measurement of cyclonic flow null angles was conducted before the first test run, but full traverses were not made for the subsequent runs. At the Outlet site, there was no traverse point with an angle greater than 10 degrees. At the Inlet site, there was only one traverse point with an angle greater than 10 degrees (Port B, point 3, 11 degrees). For subsequent runs, spot checks were made at the three points with the highest measured angles, to make sure that the angles had not increased. No angles greater than 10 degrees were measured during the spot checks.

Otherwise, the sampling, sample recovery, and sample shipping were conducted according to the test protocol.

The data reduction and validation procedures were conducted according to the test protocol. Data entries and spreadsheet calculations were verified. Calibration data were checked and verified. The chain of custody was verified for all samples.

Laboratory reports were reviewed for QA performance. Duplicate analyses were conducted as required, to determine analytical repeatability. The RPD's were within the 10% criteria, except one sample that was 21%. In that case, the higher value was used in calculation of the test results.

The results for Run 2-Hg-In show non-detection, but the detection limit for that run was higher than the detected amounts for Runs 1 and 3. The variation in the detection limits was due to the variation in the amount of particulate material collected on the filters. Since the laboratory digested an aliquot from the total mass of material, a larger mass calculates to a larger detection limit. Note that while the total amount of filterable or "front-half" particulate was similar from one run to another, Run 2 had more on the filter and less in the probe wash as compared to the other runs.

The results were calculated without correction for the solution blanks. The blank corrections were deleted because there was no detection of Mercury in any of the solution blank samples. The "blank-corrected" results shown on the spreadsheet were therefore not used for presentation of the test results.

Kevin J. Crosby



PROJECT #: 99057 PROJECT MANAGER: Erick Mirabella

SAMPLING METHOD: Ontario Hydro QA OFFICER: Kevin Corby

CLIENT/LOCATION: Air Products / Stockton Cogen ANALYTICAL METHOD: C1AA

group

vegetable

A

QUALITY CONTROL CHECKLIST Ontario Hydro Mercury Speciation

Quality Assurance Officer: Kevin J. Crosby

Calibration and Maintenance Data

Instrument Type	Maintenance or Corrective Action	Instrument Calibration	Method of Calibration or Comparison Standard	Acceptance Limits
Dry Gas Meter (Inlet)	calibration	pre-test / post-test	calibrated dry test meter	± 2% of volume measured
Dry Gas Meter (Outlet)	calibration	pre-test / post-test	calibrated dry test meter	± 2% of volume measured
S-Type Pitot Tubes	calibration	pre-test / post-test	EPA Method 2 (vine tunnel)	EPA Method 2, 2%, 5%
Vacuum / Pressure Gauges	comparison	pre-test	calibrated manometer	± 3% iwgt
Field Barometer	calibration (airport)	pre-test	mercury barometer	± 0.2" Hg
Thermocouples	calibration	pre-test / post-test	NBS mercury thermometer	± 4 °F for <400 °F
Temperature Devices	comparison	pre-test	precision potentiometer	± 2% full scale reading
Probe Nozzles	Clean and inspect	pre-test	with micrometer	≤ ± 0.10 mm, 3 measurements
Continuous Analyzers	calibration	before each usage	EPA NBS gas reference methods	<2%, 3%, 5% as in CARB 100
Pitot Lines	leak check	pre-test / post-test	with manometer	± 2% iwgt
Pumps	leak check	pre-test	with vacuum gauge	± 0.2" Hg each test
Sample Lines	new Teflon	pre-test	clean w/ water - also acid	N/A
Glassware, probes	clean and inspect	pre-test	clean w/ acid, citronox, water	N/A

Checked with whom	Date of Completion	Initials
EM/PG	10-15-99	KJC
EM/PG	10-15-99	KJC
EM	10-19-99	KJC
EM	10-19-99	KJC
EM	10-19-99	KJC
EM	10-19-99	KJC
EM	10-19-99	KJC
EM	10-19-99	KJC
DD/PG	10-20-99	KJC
EM	10-20-99	KJC
JP, PG	10-22-99	KJC
JP, PG	10-22-99	KJC
EM	10-15-99	KJC
DD/PG	10-15-99	KJC

Equipment Data

preliminary stack measurements (inlet) traverse points, port couplings
preliminary stack measurements (outlet) traverse points, port couplings
calculate nozzle size and sampling factor for both locations
make sure vacuum grease is not used on sample train
make sure sample train and pitots are leak checked before and after tests
make sure oven temperatures are maintained within 27 °F of the flue gas temperatures

JP, PG	10-19-99	KJC
JP, PG	10-19-99	KJC
DD/PG	10-19-99	KJC
JP, PG	10-20-99	KJC
JP, PG	10-20-99	KJC
JP, PG	10-20-99	KJC

also checked Run 2+3
on 10-21-99
and 10-22-99 KJC

Field Data

O₂ / CO₂ comparison with plant data and past report data
stack flow comparison with plant data and past report data
check sampling data
calculate sampling data averages
verify computer spreadsheets
verify data input to spreadsheets

EM/PG	10-20-99	KJC
EM	10-20-99	KJC
EM	10-20-99	KJC
EM	10-20-99	KJC
EM	10-15-99	KJC
EM	1-19-2000	KJC

Verified 1-19-2000 KJC
verified 1-19-2000 KJC

Other

make sure field and reagent blanks are taken
make sure permanganate impingers contents are not becoming bleached during test run

DD/PG	10-20-99	KJC
JP, PG	10-20-99	KJC

also checked Run 2+3
on 10-21-99 KJC
and 10-22-99 KJC

Quality Control Checklist Ontario Hydro Mercury Speciation

Quality Assurance Officer:

Kevin J. Crosby

10. Sampling

traverse points - location
same for velocity and sampling?
nozzle diameter
sampling time and volume

Specification	Actual	Comments
EPA Method 1		Inlet 4 ports, 6 pts. / Outlet 16 pts.
—	—	yes
For 1.0 to 2.5 dscm sample - Isokinetic		
"		Outlet - 2.2 dscm
		Inlet - 1.5 dscm

11. Preparation of Apparatus

filter weights
glassware cleaning
assembly of train as in Figure 1
proper reagents in impingers
impinger weights
grease used? How keep leak-free?
nozzle attachment
traverse points marked on probe
leak check train, pre and post-test
leak check pitot, pre and post-test

Specification	Actual	Comments
$\pm 0.1 \text{ mg}$	$\pm 0.1 \text{ mg}$	
EPA Spec.	Alconox, 50% HNO_3	Citronox, DI water
See Fig 1	As in Fig-1	
"	yes	
$\pm 0.1 \text{ gm}$	$\pm 0.1 \text{ gm}$ on-site	
no grease	no grease - ground glass joints sealed OK	
non-contaminating	ground-glass taper joint	
tape or other marks	"white-out" marker	
$< 0.02 \text{ cfm}$	All OK	
$> 3 \text{ in. hold for 15 sec.}$	All OK	

13. Procedures

isokinetic
 120°C or within 15°C of stack temp
impingers less than 20°C
how handle point and port changes?

any filter changes?
run data recorded properly?
probe leak check
pitot leak check

Recovery

cap all ends and move to field lab

container 1

container 2

container 3

KMnO_4 added until purple?

container 4

rinses all OK?

container 5

rinses all OK?

container 6 - silica gel

Specification	Actual	Comments
$\pm 10\%$	All within 10%	
←	Yes	
←	Yes	
As quickly as possible	Outlet - move point, take readings, set ΔH - 2 people for port change - leak check, disassemble,	
allowed w/ leak check	none	move, reassemble, leak check
complete form	yes	Inlet - similar except 4 people
$< 0.02 \text{ cfm}$	all good	to change ports, no disassembly,
$> 3 \text{ in. hold 15 sec.}$	all good	keep pump running during port change.
Cap, transport	Cap w/ parafilm	
Filter to petri dish		
Rinse w/ 0.1N HNO_3		
Rinse "		
Add KMnO_4 , rinse 10% HNO_3 , find 0.1N HNO_3		
rinse 0.1N HNO_3		
rinse 0.1N HNO_3 , Hydroxylamine Sulfate		
collect or weigh	weighed entire impinger	

All collected
and rinsed as
described in
Method.

Measured volumes of
rinses and impinger
contents and any
additions to samples

KJC

Quality Control Checklist

Ontario Hydro Mercury Speciation

Quality Assurance Officer:

Kevin J. Crosby

12. Calibration for Method

nozzle diameter
pitot tube
dry gas meter
orifice meter
probe heater
thermocouple
temp. readout
leak check meter system
barometer

Value	Cal Date	Comments
Inlet 4.24 mm (0.171 in.)		
3.94 mm (0.155 in.)	10-20-99	Micro meter - avg. 3 readings
0.84	10-16-99	Post-test on 10-22-99
Method 5, Section 5.3	7-6-99, 6-18-99	Post-test cals on 10-25-99
"	"	"
± 27°F of stack temp	Checked on-site	during each run
1.5% of temp within	10% of absolute stack temp.	- Cal 10-14-99 - Post test
Hold 5 to 7 inches	OK for each test	on 10-25-99
± 0.1 in. Hg	10-20-99	Checked vs. Stockton Airport, 1/2 mile away.

Note - verified each test day 1/2

7. Apparatus

nozzle
probe
pitot tube
diff P gauges
filter holder
umbilical tube
heating system
impinger train
extra impinger
for high moisture?
metering system
barometer
temperature gauges
pressure gauges

Specification	Actual	Comments
Glass	Glass, taper joint ground glass	
Glass	Glass	
Type 1	Type 8	
manometers	manometers	
Glass	Glass	
Heated PTFE	Heated Teflon-PTFE	> 120°C
Within 27°F of stack	Commoned Yes -	within 27°F of stack temp.
8 impingers	8 impingers	
—	—	Not used - moisture < 10% vol.
See Method, 7.1.9	Method 5 type	Calibrated, leak-checked
± 0.1 in. Hg	± 0.01 in. Hg	Checked vs. adjacent airport
attached to pitot	Thermocouple attached to pitot	Calibrated
manometer	Pitot turned to null angle, manometer	

8. Reagents

filters
KCl solution
HNO3/H2O2 absorbing sol.
H2SO4/KMnO4 absorbing sol.
HNO3 rinse sol.
10% HNO3 rinse sol.
10% hydroxylamine rinse sol.
KMnO4 recovery sol.

Specification	Actual	Comments
Quartz fiber	2500 QAT-UP Pall	
(Made from reagent)	Made on-site each day	
grade chemicals	using trace-metal grade	
	reagent chemicals	

KJC

Quality Control Checklist

Ontario Hydro Mercury Speciation

Quality Assurance Officer:

Kevin J. Crosby 10-20-99

Chain of Custody

stack samples
prep of apparatus
transport
sampling
transport
recovery
packing
shipping
laboratory
fuel samples
sampling
transport
packing
shipping
laboratory

Design Specification	Indicator Actual	Comments
Inlet	Outlet	
DD, EM	DD, EM	
EM, JP	EM, PG	carried to site
JP, KC, DD	PG, EM	manipulated for sampling
JP, EM	EM, PG	carried to field lab
DD	DD	} in field lab
DD	DD	
DD	DD	} locked overnight
DD	DD	
DD	DD	} drove samples to laboratory
All by Air Products / Stockton		Cogen personnel

Blanks Taken

Field Blank
Reagent Blanks

[illegible]

Quality Control Checklist

Ontario Hydro Mercury Speciation

Quality Assurance Officer:

Kevin J. Crosby 10-21-99

Chain of Custody

stack samples
prep of apparatus
transport
sampling
transport
recovery
packing
shipping
laboratory
fuel samples
sampling
transport
packing
shipping
laboratory

<u>Inlet</u> <u>Specification</u>	<u>Outlet</u> <u>Actual</u>	<u>Comments</u>
DD, EM	DD, EM, PG	
KC, JP	EM, PG	carried to site
JP, KC, DD	PG, EM	sampling
JP, KC	EM, PG	carried to field lab
DD	DD	} in field lab
DD	DD	
DD	DD	} locked overnight
DD	DD	
		} drove samples
		} to laboratory
All by Air Products / Stockton Oxygen personnel		

Blanks Taken

Field Blank
Reagent Blanks

[illegible]

Quality Control Checklist

Ontario Hydro Mercury Speciation

Quality Assurance Officer:

10-22-99 Kevin J. Crosby

Chain of Custody

- stack samples
- prep of apparatus
- transport
- sampling
- transport
- recovery
- packing
- shipping
- laboratory
- fuel samples**
- sampling
- transport
- packing
- shipping
- laboratory

Specification	Actual	Comments
Inlet	Outlet	
DD, EM	DD, EM	
KC, JP	PG, EM	carried to site
JP, KC, DD, EM	PG, EM	sampling
KC, JP	EM, PG	carried to field lab
DD	DD	2 in field lab
DD	DD	5 packed
DD	DD	3 drove directly
DD	DD	5 to laboratory
All by Air Products / Stockton		Cogen personnel

Blanks Taken

Field Blank
Reagent Blanks

[illegible]

Appendix A.6

Collection of a Gross Sample of Coal (ASTM D-2234-97a)



Standard Practice for Collection of a Gross Sample of Coal¹

This standard is issued under the fixed designation D 2234; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

INTRODUCTION

Data obtained from coal samples are used in establishing price, controlling mine and cleaning plant operations, allocating production costs, and determining plant or component efficiency. The task of obtaining a sample of reasonable weight to represent an entire lot presents a number of problems and emphasizes the necessity for using standard sampling procedures.

Coal is one of the most difficult of materials to sample, varying in composition from noncombustible particles to those which can be burned completely, with all gradations in between. The task is further complicated by the use of the analytical results, the sampling equipment available, the quantity to be represented by the sample, and the degree of precision required.

This practice gives the overall requirements for the collection of coal samples. The wide varieties of coal-handling facilities preclude the publication of detailed procedures for every sampling situation. The proper collection of the sample involves an understanding and consideration of the physical character of the coal, the number and weight of increments, and the overall precision required.

1. Scope

1.1 This practice covers procedures for the collection of a sample under various conditions of sampling. The sample is to be crushed and further prepared for analysis in accordance with Method D 2013. However, the procedures for dividing large samples before any crushing are given in this practice.

1.2 This practice describes general and special purpose sampling procedures for coals (1) by size and condition of preparation (for example, mechanically cleaned coal or raw coal) and (2) by sampling characteristics.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

- 2.1 *ASTM Standards*:
D 121 Terminology of Coal and Coke
D 2013 Method of Preparing Coal Samples for Analysis²
E 177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods³
E 456 Terminology Relating to Quality and Statistics²

3. Terminology

3.1 Definitions of Terms Specific to This Standard:

3.1.1 accuracy:

3.1.1.1 *generally*—a term used to indicate the reliability of a sample, a measurement, or an observation.

3.1.1.2 *specifically*—a measure of closeness of agreement between an experimental result and the true value. Example: the observed and true sulfur content of a coal consignment. This measure is affected by chance errors as well as by bias.

3.1.2 *gross sample*—a sample representing one lot of coal and composed of a number of increments on which neither reduction nor division has been performed.

3.1.3 *increment*—a small portion of the lot collected by one operation of a sampling device and normally combined with other increments from the lot to make a gross sample.

3.1.4 *representative sample*—a sample collected in such a manner that every particle in the lot to be sampled is equally represented in the gross or divided sample.

3.1.5 *sample*—a quantity of material taken from a larger quantity for the purpose of estimating properties or composition of the larger quantity.

3.1.6 *size consist*—the particle size distribution of a coal.

3.1.7 *random variance of increment collection (unit variance)*, S_r^2 —the theoretical variance calculated for a uniformly mixed lot and extrapolated to 0.5-kg (1-lb) increment size. For a method of estimating this variance, see Annex A1.

3.1.8 *segregation variance of increment collection*, S_s^2 —the variance caused by nonrandom distribution of ash content or other constituent in the lot. For a method of estimating this variance, see Annex A1.

3.1.9 *total variance*, S_o^2 —the overall variance resulting from collecting single increments, and including division and analysis of the single increments. For a method of estimating this variance, see Annex A2.

4. Summary of Practice

4.1 The general-purpose sampling procedures are in-

¹ These methods are under the jurisdiction of ASTM Committee D-5 on Coal and Coke and are the direct responsibility of Subcommittee D05.23 on Sampling. Current edition approved Dec. 10, 1997. Published February 1998. Originally published as D 2234 - 63 T. Last previous edition D 2234 - 97.

² *Annual Book of ASTM Standards*, Vol 05.05.

³ *Annual Book of ASTM Standards*, Vol 14.02.

tended to provide, in 19 of 20 cases, dry ash results that are within an interval of $\pm 1/10$ of the average dry ash results that would be obtained in hypothetical repeated sampling.

4.2 Special-purpose sampling procedures apply to the sampling of coal when other precision limits are required, or when other constituents are used to specify precision, or for performance tests.

4.3 For coals of known size and condition of preparation, tables are given for the determination of the number and weight of increments required for a gross sample for both general and special-purpose sampling. For coals having known sampling characteristics, as determined by the use of appropriate test and statistical procedures given in this practice, the number and weight of the increments required for either general purpose or special-purpose precision can be determined.

4.4 The procedures appear in the following order:

Test Method	Section
Sampling of Coals Based on Size and Condition of Preparation	8.1
General-Purpose Sampling Procedure	8.1.1
Number and Weight of Increments	8.1.1.2
Number of Gross Samples	8.1.1.4
Special-Purpose Sampling	8.1.2
Number and Weight of Increments	8.1.2.2
Number of Gross Samples	8.1.2.3
Sampling of Coals Based on Known Sampling Characteristics	8.2
Principles of Sampling by Sampling Characteristics	8.2.1
General-Purpose Sampling	8.2.2
Number and Weight of Increments	8.2.2.1
Number of Gross Samples	8.2.2.2
Special-Purpose Sampling	8.2.3
Number and Weight of Increments and Number of Gross Samples	8.2.3.2
Division of the Gross Samples Before Crushing	8.3
Sampling of Coal for Total Moisture Determination	8.4
Types of Moisture Samples	8.4.1
Entire Gross Samples	8.4.1.1
Special Moisture Subsamples	8.4.1.2
Other Subsamples for Moisture Testing	8.4.1.3
Special Precautions	8.4.2
Weight of Increments	8.4.3
Number of Increments	8.4.4
Moisture Sampling Based on Known Sampling Characteristics	8.4.4.1
Moisture Sampling Based Only on Size	8.4.4.2

5. Significance and Use

5.1 It is intended that this practice be used to provide a representative sample of the coal from which it is collected. Because of the variability of coal and the wide variety of sampling equipment, caution should be used in all stages of sampling from system specifications and equipment procurement to equipment acceptance testing and actually taking the final sample.

5.2 After further processing (Method D 2013), the sample may be analyzed for a number of different parameters. These parameters may affect the lot's value, its ability to meet specifications, its environmental impact, as well as other properties.

6. Increment Collection Classification

6.1 The type of selection, the conditions under which individual increments are collected, and the method of spacing of increments from the coal consignment or lot are classified according to the following descriptions and Table 2. These designations are to be used for sampling specifica-

tions and for descriptions of sampling programs and sampling equipment.

6.2 *Types of Increments*—The types of selection of increments are based on whether or not there is human discretion in the selection of the pieces of coal or portions of the coal stream.

6.2.1 *Type I*, in which specific pieces or portions are not subject to selection on a discretionary basis. This includes that in which the increment is collected in precise accord with previously assigned rules on timing or location that are free of any bias. Type I selection increments generally yield more accurate results.

6.2.2 *Type II*, in which some measure of human discretion is exercised in the selection of specific pieces of coal or of specific portions of the stream, pile, or shipment.

6.3 *Conditions of Increment Collection*—The conditions under which individual increments are collected are the conditions of the main body of coal relative to the portion withdrawn. Four conditions are recognized:

6.3.1 *Condition A (Stopped-Belt Cut)*, in which a loaded conveyor belt is stopped and a full cross-section cut with parallel sides is removed from the coal stream. The distance between the parallel faces shall not be less than three times the normal top size of the coal.

6.3.2 *Condition B (Full-Stream Cut)*, in which a full cross-section cut is removed from a moving stream of coal.

6.3.3 *Condition C (Part-Stream Cut)*, in which a portion, not a full cross section, is removed from a moving stream of coal.

6.3.4 *Condition D (Stationary Coal Sampling)*, in which a portion of coal is collected from a pile, a rail car, a barge, or a shiphold.

6.4 *Spacing of Increments*—The spacing of increments pertains to the kind of intervals between increments. Two spacing methods are recognized: systematic and random. Systematic spacing is usually preferable.

6.4.1 *Systematic Spacing 1*, in which the movements of individual increment collection are spaced evenly in time or in position over the lot.

6.4.2 *Random Spacing 2*, in which the increments are spaced at random in time or in position over the lot.

7. Organization and Planning of Sampling Operations

7.1 *Precaution*—It is imperative that every gross sample be collected carefully and conscientiously and in strict accordance with the procedures prescribed in this practice; for if the sampling is done improperly, the sample will be in error, and it may be impossible or impracticable to take another sample. However, if the analysis is in error, another analysis can easily be made of the original sample, except for moisture.

7.2 *Selection of Appropriate Sampling Procedure*—Variations in coal-handling facilities make it impossible to publish rigid rules covering every sampling situation in complete and exact details. Proper sampling involves an understanding and proper consideration of the minimum number and weight of increments, the size consist of the coal, the condition of preparation of the coal, the variability of the constituent sought, and the degree of precision required.

7.2.1 *Number and Weight of Increments*—The number

TABLE 1 Increment Types, Conditions, and Spacing

Condition of Increment Collection from the Main Body of Coal	Types of Increment			
	Type I No Human Discretion Is Used		Type II Human Discretion Is Used	
	Spacing of Increments		Spacing of Increments	
	1. Systematic	2. Random	1. Systematic	2. Random
Condition A, stopped belt cut	I-A-1	I-A-2	II-A-1	II-A-2
Condition B, full-stream cut	I-B-1	I-B-2	II-B-1	II-B-2
Condition C, part-stream cut	I-C-1	I-C-2	II-C-1	II-C-2
Condition D, stationary sampling	I-D-1	I-D-2	II-D-1	II-D-2

and weight of increments required for a given degree of precision depends upon the variability of the coal. This variability increases with an increase in free impurity. A coal high in inherent impurity and with comparatively little free impurity may exhibit much less variability than a coal with a low inherent impurity and a relatively high proportion of free impurity. For most practical purposes, an increase in the ash content of a given coal usually indicates an increase in variability. It is imperative that not less than the minimum specified number of increments of not less than the minimum specified weight be collected from the lot. For Condition D, the increments shall be of equal weight.

7.2.2 Increment Collection Method to Be Used—To obtain complete representation of all sizes, it is most desirable that the sample increments be withdrawn from the full cross section of the stream. The best possible increment is a full cross-section cut removed from a stopped belt, Classification I-A-1 in Table 1. The best possible increment from a flowing stream of coal is one obtained by moving a cutter device entirely across the stream at a uniform speed, the same for each increment, into one side of the stream and out of the other, without allowing the receptacle to overflow (Classification I-B-1 in Table 1). Classification methods given in Table 1 are listed in order of decreasing reliability. The highest possible classification method, wherever feasible, should be used. Details of sampling procedures should be agreed upon in advance by all parties concerned. Whenever circumstances dictate utilization of increment collection classifications "Condition C" or "Condition D" or "Type II," details of sampling procedure shall be agreed upon in advance by all parties concerned.

7.3 Distribution of Increments—It is essential that the increments be distributed throughout the lot to be sampled. This distribution is related to the entire volume of the lot, not merely its surface or any linear direction through it or over it. If circumstances prevent the sampler from applying this principle, the lot is sampled only in part, and the gross sample is representative only of this part. The spacing of the increments shall be varied if the possibility exists that increment collection may get "in phase" with the sequence of coal variability. Example: routine sampling of commercial coal from a continuous stream (conveyor belt) in which increment collection is automatic and its sequence coincides with the "highs" or "lows" in the content of fines.

7.4 Dimensions of Sampling Device—The opening of the sampling device shall be at least $2\frac{1}{2}$ to 3 times the top size of the coal. For practical reasons, however, it is recommended that the opening of any sampling device be not less than 31.8 mm ($1\frac{1}{4}$ in.), regardless of the top size of the coal. The

sampling device shall be of sufficient capacity to retain completely or pass entirely the increment without loss or spillage.

7.5 Movement of Sampling Device—In sampling from moving streams of coal, the sampling device shall be designed to minimize disturbance of the coal, thereby avoiding separation of various coal densities or sizes or both. To prevent segregation and rejection as a result of disturbance of the coal stream, practical evidence indicates that the velocity with which the cross-stream cutting instrument travels through the stream should not exceed 18 in./s [457 mm/s]. Cutters operating in excess of 18 in./s are available. However, the user should be aware that the cutting device must be proven to be free of bias under the normal range of conditions expected.

7.6 Preservation of Moisture—The increments obtained during the sampling period shall be protected from changes in composition as a result of exposure to rain, snow, wind, sun, contact with absorbent materials, and extremes of temperature. The circulation of air through equipment must be reduced to a minimum to prevent both loss of fines and moisture. Samples in which moisture content is important shall be protected from excessive air flow and then shall be stored in moisture-tight containers. Metal cans with airtight lids, or heavy vapor-impervious bags, properly sealed, are satisfactory for this purpose.

7.7 Contamination—The sampling arrangement shall be planned so that contamination of the increments with foreign material or unrelated coal does not create bias of practical consequence.

7.8 Mechanical System Features—With mechanized systems, it is essential that the system as a whole, including the sample cutter, chutes, conveyors, crushers, and other devices be self-cleaning and nonclogging and be designed in a manner that will minimize the need for maintenance.

7.9 Personnel—Because of the many variations in the conditions under which coal must be sampled, and in the nature of the material being sampled, it is essential that the samples be collected under the direct supervision of a person qualified by training and experience for this responsibility. Where human labor is employed to collect the increments, it is essential that samples be collected by a trained and experienced sampler or under the direct personal observation of such a person. This includes sampling for the purpose of determining sampling characteristics of a coal or characteristics of a particular sampling apparatus.

7.10 Criteria of Satisfactory Performance—A satisfactory sampling arrangement is one that takes an unbiased sample

at the desired degree of precision of the constituent for which the sample is to be analyzed. One fundamental characteristic of such an arrangement is that the size consist of the sample will adequately represent the true size consist of the coal. Sampling systems shall be tested initially and at regular intervals to determine whether the sample adequately represents the coal. In addition, sampling systems should be given a rough performance check as a matter of routine. This is done by comparing the weight or volume of collected sample with that of the total flow of coal to ensure a constant sampling ratio.

7.11 Relative Location of Sampling and Weighing—It is preferable that coal be weighed and sampled at the same time. If there is a lapse in time between these two events, consideration should be given by both the purchaser and the seller to changes in moisture during this interval and the consequent shift in relationship of moisture to the true quality of the coal at the instant when ownership of the coal transfers from one to the other.

8. Procedures

8.1 Sampling of Coals Based on Size and Condition of Preparation:

8.1.1 General-Purpose Sampling:

8.1.1.1 The general-purpose sampling procedures are intended to provide, in 19 of 20 cases, dry ash results that are within the interval of $\pm 1/10$ of the average dry ash results that would be obtained in hypothetical repeated sampling. Under some conditions, as detailed in 7.2.1 and 7.2.2, Conditions C and D and Type II, this precision may not be obtained.

8.1.1.2 Number and Weight of Increments—Obtain the number and weight of increments as specified in Table 2 except as provided in 8.1.1.5(b). Determine the minimum number of increments from the condition of preparation, and determine the minimum weight of each increment from the top size of the coal. Classify the coals to be sampled according to the general purpose procedure into three groups by top size. Further classify each of these groups into two subgroups in accordance with the condition of preparation. These classifications are shown in Table 2.

8.1.1.3 Variations in construction of the sampling device and flow, structure, or size consist of the coal may make it impracticable to collect increments as small as the minimum weight specified in Table 2. In such cases, collect an increment of greater weight. However, do not reduce the minimum number of increments, regardless of large excesses

of individual increment weights. Table 2 lists the absolute minimum number of increments for general-purpose sampling which may not be reduced except as specified in 8.1.1.5(b). Other considerations may make it advisable or necessary to increase this number of increments.

8.1.1.4 Number of Gross Samples—Under the general-purpose sampling procedure, for quantities up to approximately 1000 tons [908 metric tons] [908 Mg] it is recommended that one gross sample represent the lot. Take this gross sample in accordance with the requirements prescribed in Table 2.

8.1.1.5 For quantities over 1000 tons [908 Mg], use any of the following alternatives:

(a) Take separate gross samples for each 1000-ton [908-Mg] lot of coal or fraction thereof.

(b) Use one gross sample to represent the total tonnage provided the number of increments, as stated in Table 2, are increased as follows:

$$N_2 = N_1 \sqrt{\frac{\text{total lot size (tons or Mg)}}{1000 \text{ tons or } 908 \text{ mg}}} \quad (1)$$

where:

N_1 = number of increments specified in Table 2 and

N_2 = number of increments required.

For example, a 4000-ton [3632-Mg] lot will require twice the number of increments specified in Table 2. Using this technique, it is theoretically possible to collect one gross sample to represent a lot of infinite tonnage. Practical experience, however, indicates the maximum size of a lot of coal to be represented by one gross sample should not exceed 10 000 tons [9080 Mg].

(c) Take separate gross samples for each 1000-ton [908-Mg] lot of coal or fraction thereof, and thoroughly mix their No. 60 sieve size analysis samples together in proportion to the tonnage represented by each sample. Make one analysis of the composite sample.

8.1.2 Special-Purpose Sampling:

8.1.2.1 This special-purpose sampling procedure shall apply to the sampling of coal when increased precision is required, and the only knowledge of the coal is its top size and conditions of preparation.

8.1.2.2 Number and Weight of Increments—Take the same number and weight of increments per gross sample as specified in Table 2, or as specified in 8.1.1.5(b).

8.1.2.3 Number of Gross Samples—To obtain increased

TABLE 2 Number and Weight of Increments for General-Purpose Sampling Procedure^A

Top Size	¾ in. [16 mm]	2 in. [50 mm]	6 in. [150 mm] ^B
Mechanically Cleaned Coal ^C			
Minimum number of increments	15	15	15
Minimum weight of increments, lb	2	6	15
Minimum weight of increments, kg	1	3	7
Raw (Uncleaned) Coal ^C			
Minimum number of increments	35	35	35
Minimum weight of increments, lb	2	6	15
Minimum weight of increments, kg	1	3	7

^A Under Conditions C and D, see 7.2.1 and 7.2.2.

^B For coals above 6-in. [150-mm] top size, the sampling procedure should be mutually agreed upon in advance by all parties concerned.

^C If there is any doubt as to the condition of preparation of the coal (for example, mechanically cleaned coal or raw coal) the number of increments for raw coal shall apply. Similarly, although a coal has been mechanically cleaned, it may still show great variation because of being a blend of two different portions of one seam or a blend of two different seams. In such cases, the number of increments should be as specified for raw (uncleaned) coal.

precision for the final result for a given consignment, increase the number of gross samples collected from that consignment and analyze each gross sample separately, reporting the average of results. To reduce errors to one half, that is, to "double" the precision, take four times as many gross samples. Similarly, to reduce errors to one third, to "triple" the precision, take nine times as many gross samples.

8.2 Sampling of Coals Based on Known Sampling Characteristics:

8.2.1 Principles of Sampling by Sampling Characteristics:

8.2.1.1 The relationship between sampling characteristics (expressed as variances) and the number of increments which will give a desired precision (expressed as the specified variance of one gross sample) is shown as follows:

$$N_I = (s_r^2 + s_r^2/W)/(s_G^2 - s_{da}^2/P) \quad (2)$$

where:

N_I = number of increments in one gross sample,

W = weight in pounds of each increment; this is selected for convenience or by the limitations imposed by the particular mechanical sampling apparatus,

s_r^2 = random variance of a 0.5-kg (1-lb) increment; this value is obtained from the special sampling program given in Annex A1 (Note A1.1),

s_s^2 = segregation variance; this value is also obtained from the special sampling program given in Annex A1 (Note A1.1),

s_{da}^2 = variance of division and analysis. Procedures for calculating this quantity are given in Annex A2 of Method D 2013.

P = number of analysis samples (prepared independently from the same gross sample), and

s_G^2 = specified variance of one gross sample. The procedure for determining this variance is given in 8.2.1.2 and 8.2.1.3.

NOTE 2—The random variance and the segregation variance, s_r^2 and s_s^2 , are each inflated by unknown amounts of variance due to division and analysis. Since this results in an increased numerator in Eq. 2, and consequently, a larger calculated number of increments, N_I , it can be considered a "safety factor" for the sampling program. However, if too many large increments are taken for the evaluation of s_r^2 and s_s^2 , the "safety factor" may become unreasonably large.

8.2.1.2 The relationship between the specified variance of one gross sample, s_G^2 , and the precision for the result of several gross samples in one test period, expressed as the test period variance, s_T^2 , is given as follows:

$$s_T^2 = s_G^2/N_G \quad (3)$$

where:

s_T^2 = test period variance,

s_G^2 = specified variance of one gross sample, and

N_G = number of gross samples in the test period.

8.2.1.3 Figure 1 shows the relationship between variance and sampling precision ($\pm 10\%$ of a given constituent, 19

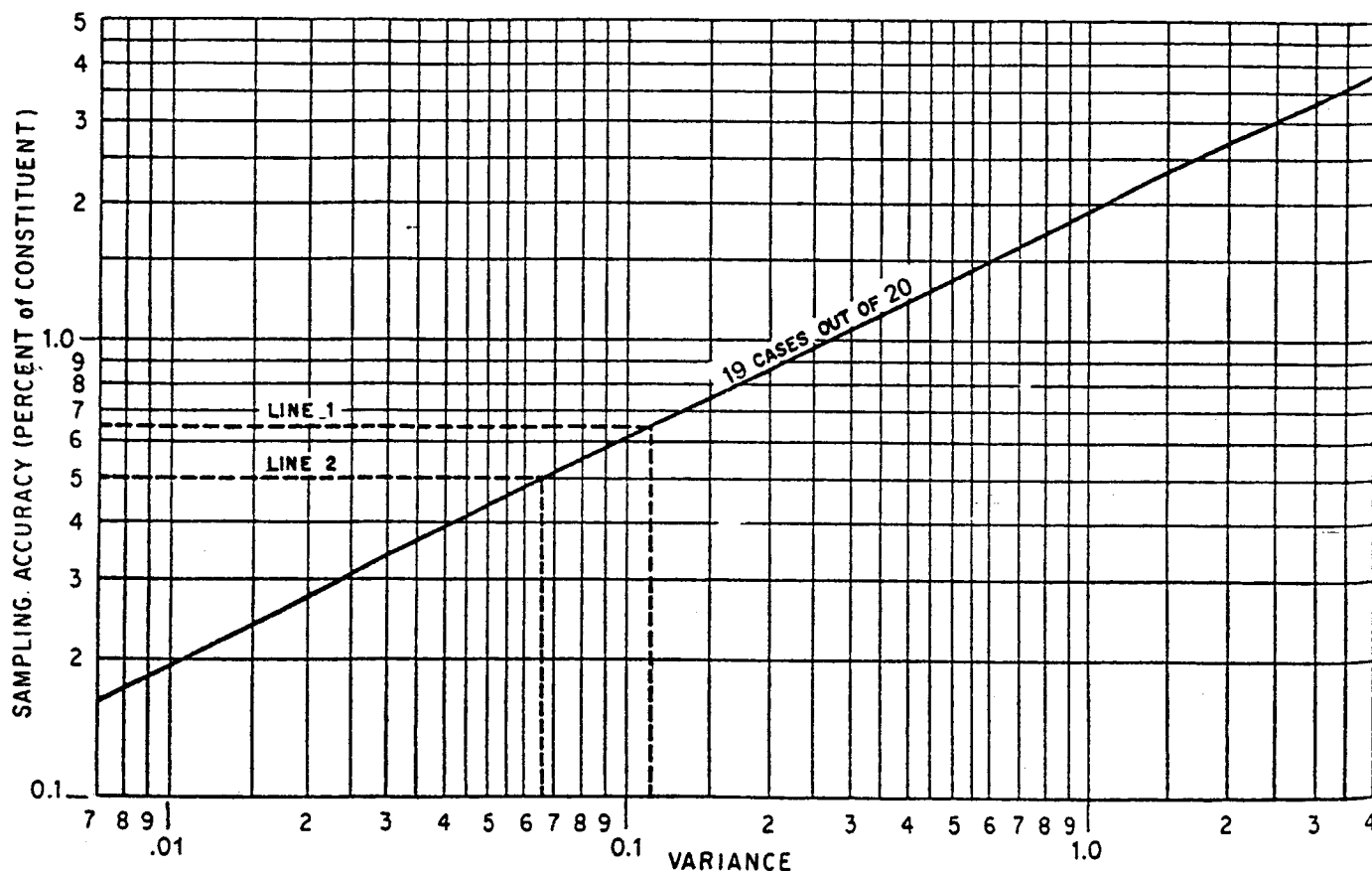


FIG. 1 Conversion of Sampling Accuracy to Variance

cases out of 20). The variance (Fig. 1) can be either the test period variance, s_T^2 , or the specified variance of one gross sample, s_G^2 . This choice will depend upon the sampling situation to be evaluated. The sampling precision (Fig. 1) can be based on any coal constituent, provided it is expressed as a percentage of that constituent. The following example is an illustration of the calculations necessary to determine the number of increments for one gross sample:

(a) *Accuracy Limits*—Assume a coal of 6.5 % average ash content. If the desired accuracy is $\pm 1/10$ of the ash content, the sampling accuracy can be expressed as ± 0.65 % ash.

(b) *Test Period Variance*—The accuracy limits given in 8.2.1.3(a) correspond to a test period variance, s_T^2 , of 0.112, from Fig. 1 (Line 1).

(c) *Specified Variance of One Gross Sample*—The specified variance of one gross sample is equal to the test period of variance, s_T^2 , multiplied by the number of gross samples in the test period, N_G (Eq 3). Assuming seven gross samples in the period, the specified variance for one gross sample is then equal to 0.112×7 or 0.784.

(d) *Number of Increments*—Assume the following information was obtained from the special sampling procedures outlined in Annex A1 of this practice and Annex A2 of Method D 2013: $s_r^2 = 12.5$, $s_s^2 = 10.2$, and $s_{da}^2 = 0.06$ (one analysis sample per gross sample). The specified variance of one gross sample, s_G^2 , as found previously, is 0.784. Further, the weight per increment for this sampling device, is found to be 23 kg (50 lb). Then, substituting into Eq 2:

$$N_I = (10.2 + 12.5/50)/(0.784 - 0.06/1) = 14.4 \text{ or } 15 \text{ increments}$$

For this coal, 15 increments of 23 kg each would be required for each gross sample, and seven gross samples would constitute the sampling period. The weighted average for the test period will be within ± 0.65 % ash, 19 cases out of 20.

8.2.1.4 The following variance relationship can be derived from Eq 2. It combines the random variance, s_r^2 , and the segregation variance, s_s^2 . This is applicable when the incremental weight is fixed by the characteristics of the sampling equipment:

$$s_T^2 = [(s_o^2 - s_{da}^2)/N_G N_I] + s_{da}^2/PN_G \quad (4)$$

where:

s_o^2 = overall variance of single increments (including division and analysis), as determined by Annex A2.

Other terms are as defined in 8.2.1.1 and 8.2.1.2. The following example demonstrates the use of the overall variance for increments, s_o^2 , in determining the number of increments for one gross sample:

(a) *Test Period Variance*—Assume a required accuracy of ± 0.5 % ash. This corresponds to a test period variance, s_T^2 , of 0.066 from Fig. 1 (Line 2).

(b) *Number of Increments*—Assume the following information was obtained from the special sampling procedures outlined in Annex A2 of Method D 2013, and Annex A2 of this practice: $s_o^2 = 3.5$ and $s_{da}^2 = 0.08$. If it is desired to take ten gross samples during the test period (N_G), with only one analysis (p) for each gross sample, the number of increments for each gross sample (N_I) can be determined by substitution into Eq 4:

$$0.066 = [(3.5 - 0.08)/(10N_I)] + [0.08/(10 \times 1)]$$

where:

$N_I = 5.9$ or 6 increments.

For this coal, six increments would be required for each of the ten gross samples. The weighted average for the test period will be within ± 0.5 % ash, 19 cases out of 20.

8.2.1.5 For sampling mixed coals, the values of random variance, segregation variance, overall variance for increments, and the variance of division and analysis for use in Eqs 2 and 4 are those obtained from special sampling programs using the mixture which is the most difficult to sample.

8.2.2 General-Purpose Sampling:

8.2.2.1 This general-purpose sampling procedure is intended for the commercial sampling of coal where the level of precision as stated in 8.1.1.1 is satisfactory to all parties involved.

8.2.2.2 *Number of Gross Samples*—Select the number of gross samples for coals of known sampling characteristics to suit the parties concerned with results from the sampling, since the number of gross samples is directly related to the establishment of the required number of increments as outlined in 8.2.1.3 and 8.2.1.4. The following factors should be remembered in selecting the number of gross samples for the test period:

(a) Too few gross samples will result in additional preparation work because of the large number of increments per gross sample which will be required.

(b) The preparation of the samples for analysis purposes will be simplified by using samples of minimum weight.

8.2.3 Special-Purpose Sampling:

8.2.3.1 Apply this special-purpose sampling procedure to the sampling of coal when other precision limits are required or when other constituents are used to specify precision.

8.2.3.2 *Number and Weight of Increments and Number of Gross Samples*—For a precision of $\pm 1/20$ of the average of all the dry ash determinations in 19 out of 20 cases when gross samples are repeatedly taken from the same lot, use Fig. 1 to determine the test period variance, s_T^2 . In this case, use the new sampling precision limitation of $\pm 1/20$ of the average dry ash in Fig. 1. Then determine the number of increments and number of gross samples as outlined in 8.2.1.3, 8.2.1.4, and 8.2.2.2.

8.2.3.3 For a precision of $\pm 1/30$ of the average ash, use Fig. 1 again to determine the test period variance, s_T^2 . In this case, use the new sampling variance, s_T^2 , precision limitations in Fig. 1.

8.2.3.4 Other precision limits may be used, or other constituents may be used to specify precision when agreed upon by the parties concerned. The principles outlined in this section will apply to all special precision limits.

8.2.3.5 Greater accuracy cannot be obtained by merely increasing the weight and number of increments if significant bias exists.

8.3 Division of the Gross Sample Before Crushing:

8.3.1 In the case of very large and unwieldy gross samples, it is permissible to divide the gross sample to reduce its weight, provided the following conditions are fulfilled:

8.3.1.1 If the entire gross sample is mixed in a suitable blender (double-cone or twin-shell tumbler) it is permissible to divide the sample using the schedule of Table 2. Test the divided sample for bias.

8.3.1.2 If each very large increment is reduced in quantity by secondary sampling, take at least six secondary increments from each primary increment. The method of collection of secondary increments must be proved to be free from bias. In no case shall the weight of a secondary increment be less than shown in the schedule of Table 2.

8.4 Sampling of Coal for Total Moisture Determinations:

8.4.1 *Types of Moisture Samples*—Moisture determinations as specified in the method to be used are to be made on the following kinds of samples.

8.4.1.1 *Entire Gross Sample*—For referee tests, air dry the entire gross sample and measure the weight loss from the entire gross sample during this drying. This procedure can be carried out on the entire gross sample as a single batch or on groups of primary increments or as separate operations on the individual primary increments; obtain, by one of these means, the total weight loss from the entire gross sample. After this air drying, the sample can be crushed or divided, or both, as required by the referee test for moisture.

8.4.1.2 *Special Moisture Subsample*—For moisture testing, a special subsample can be taken from a gross sample before any operations of air drying or crushing. Take this subsample from the gross sample in accordance with the requirements of 8.3.

8.4.1.3 *Other Subsamples for Moisture Testing*—For moisture testing, a subsample can be used that is collected after the initial crushing and dividing of a gross sample. The procedures for the crushing and dividing, and for this subsequent subsampling for moisture, are given in Method D 2013.

8.4.2 *Special Precautions*—Collect samples and subsamples for moisture in such a manner that there is no unmeasured loss of moisture of significant amount. Make adequate weighings before and after drying or other operations to measure all significant weight losses.

8.4.3 *Weight of Increments*—The minimum weight of each increment must be that which is sufficient as to be free

of bias. This depends on the top size of the coal in the stream being sampled, the dimensions of the collection device, and other factors of the withdrawal of the increment. Since much of the moisture tends to be distributed uniformly across the surface, moisture bias is present when the size consist of the sample is not the same as the size consist of the lot sampled. In addition, when there is no knowledge of the sampling characteristics for moisture, each increment shall not weigh less than the values in Table 2.

8.4.4 *Number of Increments*—The number of increments required for a given degree of precision depends on the weight of the increments, the distribution of the moisture, and the total amount of moisture. The distribution of moisture, however, is not easily evaluated independent of total moisture; consequently, the combined effects can be measured by determining the sampling characteristics for moisture.

8.4.4.1 *Moisture Sampling Based on Known Sampling Characteristics*—When the sampling characteristics for moisture are known, calculate the number of increments required for a desired degree of precision. The procedures are those given in Section 7.

8.4.4.2 *Moisture Sampling Based Only on Size*—When there is no knowledge of the sampling characteristics for moisture, collect at least the number of increments from the lot of coal as those given in Table 2. When a special moisture subsample is taken from the gross sample before any drying or crushing operations, collect the number of increments for the subsample as specified in 8.3.

9. Precision and Bias

9.1 The precision of the general-purpose sampling procedure, based on size and condition of preparation, is stated in 8.1.1.1. If a different precision is required, reference 8.1.2. The precision of sampling coals of known sampling characteristics, either general purpose or special purpose, may be estimated by following the appropriate procedure of Section 8.

ANNEXES

(Mandatory Information)

A1. TEST METHOD FOR DETERMINING THE VARIANCE COMPONENTS OF A COAL

A1.1 Scope

A1.1.1 This test method covers a procedure for determining the following variance components of a coal:

A1.1.1.1 The random variance of a 0.5-kg (1-lb) increment, s_r^2 , and

A1.1.1.2 The segregation variance, s_s^2 , the variance caused by nonrandom distribution of the ash content in the lot or consignment.

A1.1.2 In this test method, each different coal will require a complete experiment, which involves the collection of two sets of 30 samples from a stopped conveyor belt. The first set of samples includes 30 very small samples to furnish data for the random variance; the second set includes 30 large samples to furnish data for the segregation variance. Since

one of the important components of variance is that due to segregation, it is essential that the 30 large samples be so distributed with respect to time that coverage of all subtypes of coal are represented.

A1.2 Apparatus

A1.2.1 The following equipment, in addition to that equipment normally provided for routine sampling, will be required:

A1.2.1.1 *Two-Section Belt Divider*—One of the sections should be approximately the width corresponding to three times the top size of the coal, and should trap a sample of between 2 and 9 kg (4 and 20 lb). The other section should be approximately the width corresponding to 20 times the top size of the coal and should trap a sample of between 36

TABLE A1.1 Schedule I: Sample Weights

Set Number	"A" Series, g	"B" Series, lb
1	89	117.4
2	126	117.5
3	152	123.4
4	109	90.7
5	149	101.7
6	87	89.6
7	110	107.7
8	142	110.8
9	123	123.0
10	111	106.2
11	140	116.4
12	121	96.7
13	112	109.0
14	122	106.9
15	158	99.8
16	160	87.6
17	55	88.6
18	76	92.3
19	105	93.0
20	132	99.8
21	108	106.6
22	86	124.2
23	142	127.8
24	123	111.3
25	133	111.6
26	261	107.2
27	129	106.0
28	150	102.8
29	108	97.7
30	99	107.4
Sum	3732	3180.7
Average	124.4 g, or 0.27 lb = w_1	106.0 lb or 48.1 kg = w_2

TABLE A1.2 Schedule II: Ash Results "A" Series

Set	Ash, %	Ash, %-squared
1	14.2	201.64
2	13.4	179.56
3	13.7	187.69
4	15.8	249.64
5	13.7	187.69
6	14.1	198.81
7	13.6	184.96
8	18.7	349.69
9	16.3	265.69
10	12.4	153.76
11	5.8	33.64
12	12.2	148.84
13	10.9	118.81
14	8.9	79.21
15	34.5	1190.25
16	8.7	75.69
17	7.5	56.25
18	15.7	246.49
19	21.8	475.24
20	11.8	139.24
21	12.2	148.84
22	11.8	139.24
23	7.1	50.41
24	12.8	163.84
25	14.0	196.00
26	6.3	39.69
27	12.3	151.29
28	7.2	51.84
29	13.1	171.61
30	11.3	127.69
Sum	391.8	5963.24

$$s_A^2 = [5963.24 - (391.8)^2/30]/29 = 29.2$$

and 68 kg (80 and 150 lb). The bottom edges of the divider should be shaped to conform to the surface of the conveyor belt.

A1.2.1.2 *Riffle Splitter*, with slots at least 2½ to 3 times as wide as the maximum size of the particles, or a manual divider and canvas for subdividing the small samples by hand.

A1.3 Procedure

A1.3.1 The following sampling procedure should be used

for each of the two required sets of samples:

A1.3.1.1 Stop the loaded belt and insert the belt divider with the division plates perpendicular to the direction of belt movement. Scrape off the coal from each section, and put each section into a separate completely labeled container. The container holding the coal from the small section of the belt divider should be labeled "A." The container holding the coal from the large section of the belt divider should be labeled "B."

A1.3.1.2 Collect a subsample from the "A" section by

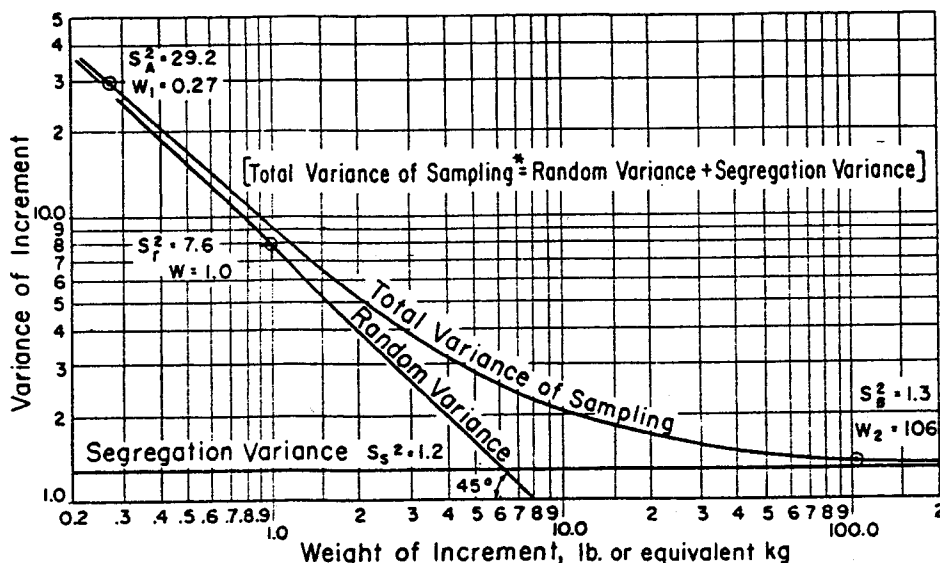


FIG. A1.1 Relation of Variance to Weight

TABLE A1.3 Schedule III: Ash Results "B" Series

Set	Ash, %	Ash, %-squared
1	13.6	184.96
2	13.2	174.24
3	14.3	204.49
4	15.3	234.09
5	15.0	225.00
6	14.3	204.49
7	13.6	184.96
8	14.7	216.09
9	14.2	201.64
10	12.5	156.25
11	13.0	169.00
12	14.3	204.49
13	13.7	187.69
14	12.8	163.84
15	13.2	174.24
16	14.0	196.00
17	10.5	110.25
18	13.5	182.25
19	15.4	237.16
20	15.0	225.00
21	14.4	207.36
22	12.8	163.84
23	13.0	169.00
24	13.0	169.00
25	12.3	151.29
26	13.1	171.61
27	14.2	201.64
28	11.6	134.56
29	13.1	171.61
30	11.4	129.96
Sum	405.0	5506.00

$$s_B^2 = \frac{[5506.00 - (405)^2/30]}{29} = 1.3$$

then:

$$S_r^2 = \frac{[0.27 \times 106 (29.2 - 1.3)]}{106 - 0.27} = 7.6$$

and

$$s_s^2 = 1.3 - (7.6/106) = 1.2$$

riffing or by manual subdivision after spreading the sample evenly on a smooth flat surface. Tag the subsample with a label "A", and weigh to the nearest gram. The weight of the subsample should be between 100 to 200 g.

A1.3.1.3 Dry the "A" subsample, grind to minus No. 60

sieve size, and determine the ash content to the nearest 0.1 %, dry basis.

A1.3.1.4 Weigh the entire "B" section, dry, and work down to an analysis sample. Determine the ash content to the nearest 0.1 %, dry basis.

A1.4 Calculation

A1.4.1 Calculate the variance of the "A" and "B" series (Note A1.1) as follows:

$$\text{Variance} = (\Sigma x^2 - (\Sigma x)^2/n)/(n - 1) \quad (5)$$

where:

Σx^2 = sum of the squares of ash results,

$(\Sigma x)^2$ = square of the sum of ash results, and

n = number of individual ash results in the series.

A1.4.2 The random variance, s_r^2 , is found from:

$$s_r^2 = [W_1 W_2 (s_A^2 - s_B^2)]/(W_2 - W_1) \quad (6)$$

where:

W_1 = average weight of small samples, lb or equivalent kg,

W_2 = average weight of large samples, lb or equivalent kg,

s_A^2 = variance of small, "A" samples, and

s_B^2 = variance of large "B" samples.

A1.4.3 The segregation variance, s_s^2 , is found from:

$$s_s^2 = s_B^2 - s_r^2/W_2 \quad (7)$$

NOTE A1.1—An actual example illustrating the treatment of data from this sampling experiment is given in Tables A1.1 to A1.3 and in Fig. A1.1.

A1.4.3.1 Using log-log paper, plot the point corresponding to an increment weight $w = 0.5$ kg (1 lb) and variance $s_s^2 = 7.6$; draw a straight line through this point, downward at 45°. This line gives the random component of variance for an increment of any weight. Plot the point corresponding to an increment weight $w_2 = 48$ kg (106 lb) and variance $s_s^2 = 1.2$; draw a straight horizontal line through this point. This line gives the segregation component of variance for an increment of any weight.

A1.4.3.2 On Fig. A1.1, find the algebraic sum of the random component and the segregation component of variance for a number of increment weights; draw a curve through these points. This curve gives the total variance of sampling for increments of any weight, including those used in the "A" and "B" series.

A2. TEST METHOD FOR ESTIMATING THE OVERALL VARIANCE FOR INCREMENTS

A2.1 Scope

A2.1.1 This test method describes the procedure for estimating the overall variance for increments of one fixed weight of a given coal. It is applicable to mechanical sampling when there is no need to explore system and random variance components, but there is a need for obtaining the overall variance for increments (the size of increments is dictated by the sampling equipment).

A2.2 Procedure

A2.2.1 The following procedure should be used to determine the overall variance of increments:

A2.2.1.1 Collect two series of individual increments at widely spaced intervals, for example, a series of ten incre-

ments, two each day for five days, followed by a second series of ten collected in similar fashion. Both series must be from the same coal.

A2.2.1.2 Collect each increment by using as much of the equipment and procedure used in routine sampling operations as possible. Remove the individual increment from the sampling system without mixing with or contaminating by any other increment. Where possible, allow it to pass through any mechanical crusher or subsampler, or both, which is located in the system before the point of blending with other increments.

A2.2.1.3 Then weigh the individual increment (if desired for record purposes) and reduce to a laboratory sample by procedures identical as possible to those used in the routine

preparation and reduction of gross samples.

A2.2.1.4 Analyze the sample for the constituents for which the variance calculations are to be made. Usually sampling specifications are based on dry ash, but where total moisture or as-received Btu is of particular concern, the analyses should be made for these.

A2.3 Calculation

A2.3.1 For each series, compute a variance value from the analyses of the ten increments as follows:

$$s^2 = (\Sigma x^2 - (\Sigma x)^2/n)/(n - 1) \quad (8)$$

where:

- s^2 = variance value for series,
- Σx^2 = sum of squares of ash results,
- $(\Sigma x)^2$ = square of the sum of ash results, and
- n = number of individual ash results in the series.

A2.3.2 For the two series, the ratio of the larger variance to the smaller should not exceed the value given in Table A2.1, Column 2. If they differ by less than this amount, the variances are combined to give the estimated overall increment variance for the coal as follows:

$$s_o^2 = C[(s_1^2 + s_2^2)/2] \quad (9)$$

where:

- s_o^2 = probable maximum value of the overall variance for increments,
- C = factor from Table X2.2, Column 3, corresponding to the number of increments per set,

TABLE A2.1 Variance Ratio Limit Values

1	2	3
Increment per Set	Variance Ratio Limit	"C" Factor
10	3.18	1.92
20	2.17	1.53
30	1.86	1.40
40	1.70	1.33
50	1.61	1.29

$s_1^2 = s^2$ from first series, and
 $s_2^2 = s^2$ from second series.

A2.3.3 If the ratio of the larger variance to the smaller does give a greater value than the Table A2.1, Column 2 value, the two series are to be considered in a single set of increments, and another set equal to this enlarged set is to be taken. For example, if originally two sets of 10 increments were taken, these would be combined to give a set of 20. Then an additional set of 20 increments would be collected, giving two sets of 20 increments each. Variance values are computed for the two new series and the test is repeated using the appropriate factors given in Table A2.2. If these results have a ratio which is less than the appropriate value in Column 2 of Table A2.2, they are combined by using Eq 9 and used as the new variance for increments.

A2.3.4 *Example*—The example given in Table A2.2 illustrates the computation of the overall variance for increments, s_o^2 . Two series of 10 increments each are used.

TABLE A2.2 Determination of the Overall Variance for Increments

Series 1			Series 2		
Increment Number, n	Dry Ash ^a (x)	(Dry Ash) ² Σx^2	Increment Number, n	Dry Ash ^a (x)	(Dry Ash) ² Σx^2
1	4.17	17.3889	11	3.07	9.4249
2	3.62	13.1044	12	4.88	23.8144
3	1.79	3.2041	13	5.14	26.4196
4	4.37	19.0969	14	3.63	13.1769
5	4.64	21.5296	15	3.17	10.0489
6	7.03	49.4209	16	7.20	51.8400
7	6.27	39.3129	17	3.52	12.3904
8	3.91	15.2881	18	0.87	0.7569
9	6.04	36.4816	19	0.72	0.5184
10	4.18	17.4724	20	4.78	22.8484
Sum	46.02	232.2998	Sum	36.98	171.2388

^a This example involves increment weights in the approximate range from 45 to 90 kg (100 to 200 lb).

^b 10 % ash was subtracted from each of the ash results to simplify the calculations.

$$s^2 = (\Sigma x^2 - (\Sigma x)^2/n)/(n - 1)$$

Series 1:

$$s_1^2 = (232.2998 - (46.02)^2/10)/9 = 2.2795$$

Series 2:

$$s_2^2 = (171.2388 - (36.98)^2/10)/9 = 3.8319$$

Variance ratio limit from Table A2.1 = 3.18

Variance ratio for two test series:

$$s_2^2/s_1^2 = 3.8319/2.2795 = 1.68 < 3.18$$

Since the computed value for the ratio is less than 3.18, variances are combined to give an estimate of the overall variance for increments, s_o^2 :

$$s_o^2 = [1.92 (2.2795 + 3.8319)]/2 = 5.867$$

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This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 100 Barr Harbor Drive, West Conshohocken, PA 19428.

Appendix A.7

EPA Methods 1, 2, 3A, 4, and 5

Continuous Emissions Monitoring System

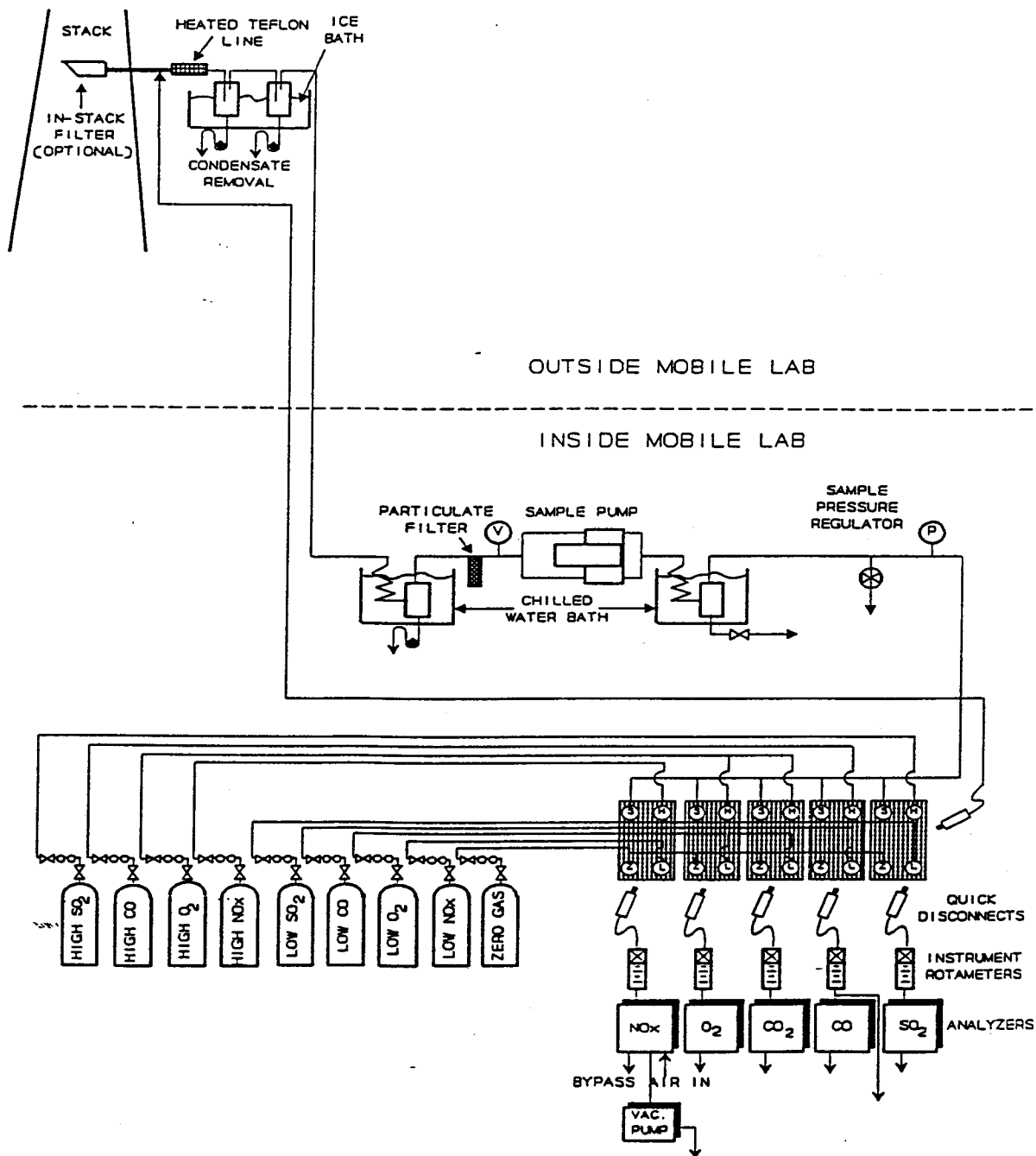
O₂, CO, CO₂, NO, NO_x and SO₂ are measured using an extractive continuous emissions monitoring (CEM) package, shown in the following figure. This package is comprised of three basic subsystems. They are: (1) the sample acquisition and conditioning system, (2) the calibration gas system, and (3) the analyzers themselves. This section presents a description of the sampling and calibration systems. Descriptions of the analyzers used in this program and the corresponding reference test methods follow. Information regarding quality assurance information on the system, including calibration routines and system performance data follows.

The sample acquisition and conditioning system contains components to extract a representative sample from the stack or flue, transport the sample to the analyzers, and remove moisture and particulate material from the sample. In addition to performing the tasks above, the system must preserve the measured species and deliver the sample for analysis intact. The sample acquisition system extracts the sample through a stainless steel probe. The probe is insulated or heated as necessary to avoid condensation. If the particulate loading in the stack is high, a sintered stainless steel filter is used on the end of the probe.

Where water soluble NO₂ and/or SO₂ are to be measured, the sample is drawn from the probe through a heated teflon sample line into an on-stack cooled (approximately 35-40°F) water removal trap. The trap consists of stainless steel flasks in a bath of ice and water. This design removes the water vapor by condensation. The contact between the sample and liquid water is minimized and the soluble NO₂ and SO₂ are conserved. This system meets the requirements of EPA Method 20. The sample is then drawn through a teflon transport line, particulate filter, secondary water removal and into the sample pump. The pump is a dual head, diaphragm pump. All sample-wetted components of the pump are stainless steel or teflon. The pressurized sample leaving the pump flows through a third condensate trap in a refrigerated water bath (≈38°F) for final moisture removal. A drain line and valve are provided to constantly expel any condensed moisture from the dryer at this point. After the dryer, the sample is directed into a distribution manifold. Excess sample is vented through a back-pressure regulator, maintaining a constant pressure of 5-6 psig to the analyzer rotameters.

The calibration system is comprised of two parts: the analyzer calibration, and the system bias check (dynamic calibration). The analyzer calibration equipment includes pressurized cylinders of certified span gas. The gases used are, as a minimum, certified to 1% by the manufacturer. Where necessary to comply with reference method requirements EPA Protocol 1 gases are used. The cylinders are equipped with pressure regulators which supply the calibration gas to the analyzers at the same pressure and flow rate as the sample. The selection of zero, span, or sample gas directed to each analyzer is accomplished by operation of the sample/calibration selector fittings.

The system bias check is accomplished by transporting the same gases used to zero and span the analyzers to the sample system as close as practical to the probe inlet. This is done either by attaching the calibration gas supply line to the probe top with flexible tubing or by actuation of a solenoid valve located at the sample conditioner inlet (probe exit). The span gas is exposed to the same elements as the sample and the system response is documented. The analyzer indications for the system calibration check must agree within 5% of the analyzer calibration. Values are adjusted and changes/repairs are made to the system to compensate for any difference in analyzer readings. Specific information on the analytical equipment and test methods used is provided in the following pages.



Schematic of CEM System

Method: Stack Gas Velocity and Volumetric Flow Rate

Reference: EPA Method 2, SCAQMD Method 2.1, ARB Method 2

Principle: The average gas velocity in a stack is determined from the measurement of the gas density and from the measurement of the average velocity head using a Type-S (Stausscheibe) Pitot tube.

Sampling Procedure: The velocity head and temperature are measured at traverse points specified by EPA Method 1 or SCAQMD Method 1.1. The velocity is measured using a Type-S Pitot tube and an inclined water manometer. The flow coefficient of the pitot tube is known. Temperature of the gas is measured using a thermocouple. The stack gas molecular weight is determined from independent measurements of O₂, CO₂, and H₂O concentrations.

Sample Analysis and Recovery: The stack gas velocity is determined from the measured average velocity head, the measured average temperature, the measured average duct static pressure, the measured dry concentrations of O₂ and CO₂, and the measured concentration of H₂O. The velocity is determined from the following set of equations:

$$V_s = 2.90 C_p \sqrt{\Delta p T_s \left[\frac{29.92}{P_s} \right] \left[\frac{28.95}{MW_{wet}} \right]} \quad [\text{ft/s}]$$

$$\Delta p = \text{Velocity/Head, inches } H_2O \quad [\text{in. } H_2O]$$

$$T_s = \text{Gas Temperature, degrees R} \quad [R]$$

$$P_s = \text{Absolute Static Pressure} \quad [\text{in Hg}]$$

$$C_p = \text{Pitot Flow Coefficient} \quad [\text{unitless}]$$

$$MW_{wet} = [(0.44)(\%CO_2) + (0.32)(\%O_2) + (0.28)(\%N_2)] \left(1 - \frac{\%H_2O}{100}\right) + (18) \left(\frac{\%H_2O}{100}\right)$$

The stack gas volumetric flow rate is determined from the measured stack gas velocity, the area of the stack at the measurement plane, and the measured gas temperature and pressure. The volumetric flow rate is determined from the following set of equations:

$$Q = (V_s)(AREA)(60) \quad [\text{wacfm}]$$

$$Q_{ws} = Q \left[\frac{T_{ref}}{T_s} \right] \left[\frac{P_s}{29.92} \right] \quad [\text{wscfm}]$$

$$Q_{sd} = Q_{ws} \left[1 - \frac{\%H_2O}{100} \right] \quad [\text{dscfm}]$$

Method:	Oxygen (O ₂) by Continuous Analyzer
Applicable Reference Methods:	EPA 3A, EPA 20, ARB 100, BA ST-14, SCAQMD 100.1
Principle:	A sample is continuously drawn from the flue gas stream, conditioned, and conveyed to the instrument for direct readout of O ₂ concentration.
Analyzer:	Teledyne Model 326A
Measurement Principle:	Electrochemical cell
Ranges:	0-5, 0-10, 0-25% O ₂
Accuracy:	1% of full scale
Output:	0-100 mV, linear
Interferences:	Halogens and halogenated compounds will cause a positive interference. Acid gases will consume the fuel cell and cause a slow calibration drift.
Response Time:	90% < 7 seconds
Sampling Procedure:	A representative flue gas sample is collected and conditioned using the CEM system described previously. If Method 20 is used, that method's specific procedures for selecting sample points are used. Otherwise, stratification checks are performed at the start of a test program to select single or multiple-point sample locations.
Analytical Procedure:	An electrochemical cell is used to measure O ₂ concentration. Oxygen in the flue gas diffuses through a Teflon membrane and is reduced on the surface of the cathode. A corresponding oxidation occurs at the anode internally, and an electric current is produced that is proportional to the concentration of oxygen. This current is measured and conditioned by the instrument's electronic circuitry to give an output in percent O ₂ by volume.
Special Calibration Procedure:	The measurement cells used with the O ₂ instrument have to be replaced on a regular basis. After extended use, the cell tend to produce a nonlinear response. Therefore, a three-point calibration is performed at the start of each test day to check for linearity. If the response is not linear (\pm 1% of scale), the cell is replaced.

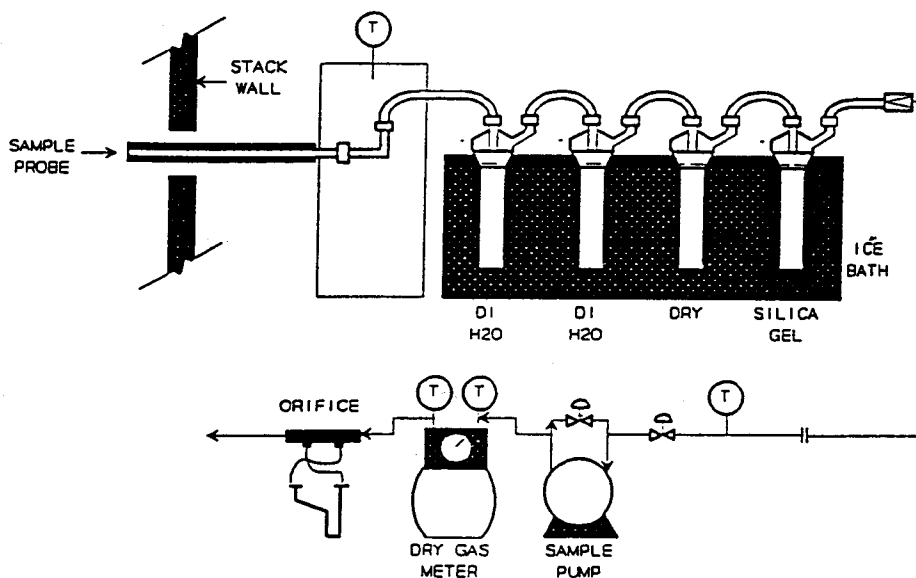
Method:	Carbon Dioxide (CO ₂) by Continuous Analyzer
Applicable Reference Methods:	EPA 3A, ARB 100, BA ST-5, SCAQMD 100.1
Principle:	A sample is continuously drawn from the flue gas stream, conditioned, and conveyed to the instrument for direct readout of CO ₂ concentration.
Analyzer:	Horiba PIR 2000
Measurement Principle:	Non-dispersive infrared (NDIR)
Accuracy:	1% of full scale
Ranges:	0-5, 0-10, 0-25%
Output:	0-10 mV
Interferences:	A possible interference includes water. Since the instrument receives dried sample gas, this interference is not significant.
Response Time:	1.2 seconds
Sampling Procedure:	A representative flue gas sample is collected and conditioned using the CEM system described previously.
Analytical Procedure:	Carbon dioxide concentrations are measured by short path length non-dispersive infrared analyzers. These instruments measure the differential in infrared energy absorbed from energy beams passed through a reference cell (containing a gas selected to have minimal absorption of infrared energy in the wavelength absorbed by the gas component of interest) and a sample cell through which the sample gas flows continuously. The differential absorption appears as a reading on a scale of 0 to 100%.

Method: **Determination of Moisture in Stack Gases**

Applicable Ref. Methods: EPA 4, ARB 1-4, SCAQMD 4.1

Principle: A gas sample is extracted at a constant rate from the source; moisture is removed from the sample stream and determined volumetrically or gravimetrically.

Sampling Procedure: The sample train used in the tests is shown in the following figure. The sample is drawn at a constant rate through a stainless steel probe. The probe is connected to an impinger train by Teflon tubing. The train consists of two Greenburg-Smith impingers which contain 100 ml water, an empty impinger as a knockout, and an impinger containing silica gel to protect the pump from moisture.



Sample Train for Determination of Moisture by EPA Method 4

Sample Recovery and Analysis:

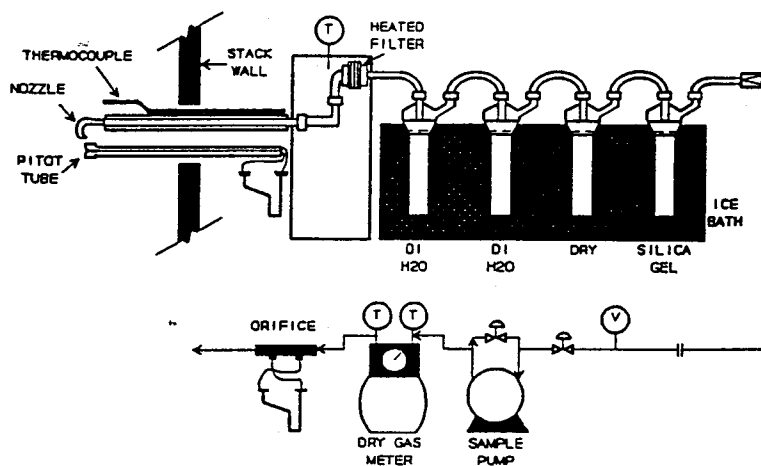
Following testing, moisture content is determined gravimetrically from initial and final impinger weights.

Method: **Total Particulate by EPA Method 5**

Reference: EPA Method 5

Principle: A metered flue gas sample is collected isokinetically and particulates are collected in a heated filter.

Sampling The sample train used in the tests is shown in the following figure. The sample is drawn isokinetically through a nozzle, a stainless steel or glass probe, and a filter in a 250°F temperature-controlled oven. This is followed by two Greenburg-Smith impingers which contain 100 ml of distilled water, an empty impinger as a knock-out, and an impinger containing silica-gel to protect the leak-tight vacuum pump and calibrated dry gas meter from moisture.



**Sample Train for Determination of Total Particulate
by EPA Method 5**

EPA Method 4 (moisture) and Methods 1 and 2 (velocity) are performed in conjunction with the test. Stack velocity is measured during the test to maintain isokinetic sampling and to measure stack flow rate. Moisture concentration is determined by weighing the impingers before and after sampling to determine the amount of moisture collected.

Sample Recovery and Analysis:

Following testing, the impingers are weighed for moisture determination and the following sample fractions are recovered:

1. Probe, nozzle and front-half glassware - acetone wash and brushing
2. Filter

The probe wash is evaporated at low temperatures, baked at 105°C, desiccated and weighed; and the filter is baked at 105°C, desiccated and weighed.

Particulate concentration is determined by dividing the mass of particulate collected by the sample gas volume.

APPENDIX B

QUALITY ASSURANCE

Appendix B.1

Quality Assurance Program Summary and ARB Certification

QUALITY ASSURANCE PROGRAM SUMMARY AND CARB CERTIFICATION

The Avogadro Group (AG) ensures the quality and validity of its emission measurement and reporting procedures through a rigorous quality assurance (QA) program. The program is developed and administered by an internal QA Officer and encompasses seven major areas:

1. Development and use of an internal QA manual.
2. QA reviews of reports, laboratory work, and field testing.
3. Equipment calibration and maintenance.
4. Chain of custody.
5. Training.
6. Knowledge of current test methods.
7. Agency certification.

Each of these areas is discussed individually below.

Quality Assurance Manual. AG has prepared a QA Manual according to EPA guidelines. The manual serves to document and formalize all of AG's QA efforts. The manual is constantly updated, and each employee involved in technical services for emission measurements is required to read and understand its contents. The manual includes details on the other six QA areas discussed below.

QA Reviews. AG's review procedure includes review of each source test report by a project QA Officer, and spot check reviews of laboratory and field work.

The most important review is the one that takes place before a test program begins. The QA Officer works closely with testing personnel to prepare and review test protocols. Test protocol review includes selection of appropriate test procedures, evaluation of any interferences or other restrictions that might preclude use of standard test procedures, and evaluation and/or development of alternate procedures.

Equipment Calibration and Maintenance. The equipment used to conduct the emission measurements is maintained according to the manufacturer's instructions to ensure proper operation. In addition to the maintenance program, calibrations are carried out on each measurement device according to the schedule outlined by the California Air Resources Board (CARB). The schedules for maintenance and calibrations are given in Tables B-1 and B-2.



Quality control checks are also conducted in the field for each test program. A partial list of checks made as part of each CEM system test series is included below as an example of the field QA procedures.

- Sample acquisition and conditioning system leak check.
- 2-point analyzer calibrations (all analyzers)
- 3-point analyzer calibrations (analyzers with potential for linearity errors).
- Complete system calibration check ("dynamic calibration" through entire sample system).
- Periodic analyzer calibration checks (once per hour) are conducted at the start and end of each test run. Any change between pre- and post-test readings are recorded.
- All calibrations are conducted using gases certified by the manufacturer to be + 1% of label value (NBS traceable).
- Calibration and CEM performance data are fully documented, and are included in each source test report.

Chain of Custody. AG maintains full chain of custody documentation on all samples and data sheets. In addition to normal documentation of changes between field sample custodians, laboratory personnel, and field test personnel, AG documents every individual who handles any test component in the field (e.g., probe wash, impinger loading and recovery, filter loading and recovery, etc.).

Samples are stored in a locked area to which only laboratory personnel have access. Neither other AG employees nor cleaning crews have keys to this area.

Data sheets are copied immediately upon return from the field, and this first generation copy is placed in locked storage. Any notes made on original sheets are initialed and dated.

Training. Personnel training is essential to ensure quality testing. AG has formal and informal training programs which include:

1. Attendance at EPA-sponsored training courses.
2. Enrollment in EPA correspondence courses.
3. A requirement for all technicians to read and understand AG's QA Manual.
4. In-house training and QA meetings on a regular basis.
5. Maintenance of training records.



Knowledge of Current Test Methods. With the constant updating of standard test methods and the wide variety of emerging test methods, it is essential that any qualified source tester keep abreast of new developments. AG subscribes to services which provide updates on EPA and CARB reference methods, and on EPA, CARB and local District rules and regulations. Additionally, source test personnel regularly attend and present papers at testing and emission-related seminars and conferences. AG personnel maintain membership in the Air and Waste Management Association and in the Source Evaluation Society.

AGENCY CERTIFICATION

AG is certified by the CARB as an independent source test contractor for gaseous and particulate measurements. AG also participates in EPA QA audit programs for Methods 5, 6 and 7.

TABLE B-1
SAMPLING INSTRUMENTS AND EQUIPMENT CALIBRATION SCHEDULE
As Specified by the CARB

Instrument Type	Frequency of Calibration	Standard of Comparison or Method of Calibration	Acceptance Limits
Orifice Meter (large)	12 months	Calibrated dry test meter	$\pm 2\%$ of volume measured
Dry Gas Meter	6 months or when repaired	Calibrated dry test meter	$\pm 2\%$ of volume measured
S-Type Pitot (for use with EPA-type sampling train)	6 months	EPA Method 2	Cp constant (+5%) over working range; difference between average Cp for each leg must be less than 2%
Vacuum Gauges Pressure Gauges	6 months	Manometer	$\pm 3\%$
Field Barometer	2 weeks (or on site)	Mercury barometer	$\pm 0.2"$ Hg
Temperature Measurement (thermocouples)	6 months	NBS mercury thermometer or NBS calibrated platinum RTD	± 4 F for <400 F $\pm 1.5\%$ for >400 F
Temperature Readout Devices	6 months	Precision potentiometer	$\pm 2\%$ full scale reading
Analytical Balance	12 months (check prior to each use)	Should be performed by manufacturer or qualified laboratory	± 0.3 mg of stated weight
Probe Nozzles	Each field day	Nozzle diameter check micrometer	Range $<\pm 0.10$ mm for three measurements
Continuous Analyzers	Every field day, Depends upon use, frequency and performance	As specified by manufacturers operating manuals, EPA NBS gases and/or reference methods	Satisfy all limits specified in operating specifications

TABLE B-2
EQUIPMENT MAINTENANCE SCHEDULE
Based on Manufacturer's Specifications and AG Experience

Equipment	Performance Requirement	Maintenance Interval	Corrective Action
Pumps	1. Absence of leaks 2. Ability to draw manufacturer required vacuum and flow	Every 300 hours of operation or 6 months, whichever is less	1. Visual inspection 2. Clean 3. Replace worn parts 4. Leak check
Flow Measuring Device	1. Free mechanical movement 2. Absence of malfunction	Every 300 hours of operation or 6 months, whichever is less After each test, if used in sampling of corrosive atmospheres (e.g. H ₂ S)	1. Visual inspection 2. Clean 3. Calibrate
Sampling Instruments	1. Absence of malfunction 2. Proper response to zero, span gas	As required by the manufacturer	As recommended by manufacturer
Integrated Sampling Tanks	Absence of leaks	Depends on nature of use	1. Steam clean 2. Leak check
Mobile Van Sampling Systems	Absence of leaks	Depends on nature of use	1. Change filters 2. Change gas dryer 3. Leak check 4. Check for system contamination
Sampling Lines	Sample degradation less than 2%	After each test or test series	Blow filtered air through line until dry

State of California
AIR RESOURCES BOARD

Executive Order G-99-026

Approval to The Avogadro Group
To Conduct Testing as an Independent Contractor

WHEREAS, the Air Resources Board (ARB), pursuant to Section 41512 of the California Health and Safety Code, has established the procedures contained in Section 91200-91220, Title 17, California Code of Regulations, to allow the use of independent testers for compliance tests required by the ARB; and

WHEREAS, it has been determined that The Avogadro Group meets the requirements of the ARB for conducting ARB Test Methods 1, 2, 3, 4, 5, 8, 17, 10, 100 for CO₂, NO_x, O₂, SO₂, and THC, and USEPA Test Method 202 pursuant to Sections 91200-91220, Title 17, California Code of Regulations when the following conditions are met;

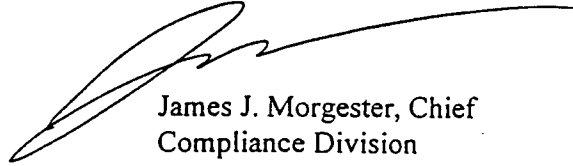
1. The Avogadro Group permanently marks or engraves an identification number on each of their pitot tubes.
2. The Avogadro Group calibrates their magnehlic gauges after each test series.
3. The Avogadro Group conducts ARB Test Method 100 for O₂ using a Teledyne 320A analyzer with either a A5 or a B1 sensor, or a paramagnetic analyzer.
4. The Avogadro Group installs a temperature gauge on the outlet of their continuous sample conditioner.
5. The Avogadro Group permanently and uniquely identify each of their probe nozzles used in isokinetic testing.
6. The Avogadro Group uses a glass frit filter support with a silicone rubber gasket for ARB Test Methods 5 and 8 unless other materials are approved by the Executive Officer.

WHEREAS, the ARB's Executive Officer pursuant to health and Safety Code section 395167 issued Executive Order G-148 delegating to the Chief of the Board's Compliance Division the authority to approve independent testers in accordance with Title 17 California Code of Regulation, Sections 91200-91220;

NOW, THEREFORE, I, James J. Morgester, chief of the Air Resources Board's Compliance Division order that The Avogadro Group is granted an approval, from the date of execution of this order, until June 30, 2000 to conduct the tests listed above, subject to compliance with Section 91200-91220, Title 17, California Code of Regulations;

BE IT FURTHER ORDERED that during the approved period the Executive Officer or his or her authorized representative may field audit one or more tests conducted pursuant to this order for each type of testing listed above.

Executed this 15TH day of APRIL 1999 at Sacramento, California.



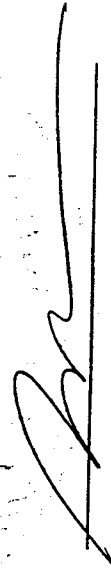
James J. Morgester, Chief
Compliance Division

State of California
Air Resources Board
Approved Independent Contractor
The Avogadro Group

This is to certify that the company listed above has been approved by the Air Resources Board to conduct compliance testing pursuant to Section 91207, Title 17, California Code of Regulations, until June 30, 2000, for those test methods listed below:

ARB Source Test Methods:

1, 2, 3, 4, 5, 8, 10, 17
100 (CO₂, NO_x, O₂, SO₂, THC)

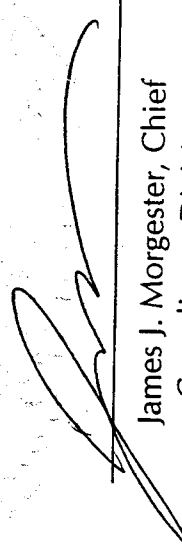


James J. Morgester, Chief
Compliance Division

State of California
Air Resources Board
Approved Independent Contractor
The Avogadro Group

This is to certify that the company listed above has been approved by the Air Resources Board to conduct compliance testing pursuant to Section 91207, Title 17, California Code of Regulations, until June 30, 2000, for those test methods listed below:

USEPA Test Method 202



James J. Morgester, Chief
Compliance Division

Appendix B.2

CEM Performance and Equipment Calibration Data

10-22-1999 12:54:16

File Name: C:\STRATA\SCHG.STR

Operator:	Erick Mirabella
Plant Name:	Stockton CoGen (Air Products)
Location:	CFBC (Baghouse Inlet & Stack
Run Length:	60 minutes
Sample Rate:	30 per minute
Average Calibration Results:	.1 minutes
Automatic Sequence, Calibration Error:	No
Automatic Sequence, System Bias:	No
Max Response Time:	Manual
Max Response Time:	1 minutes
Traverse During Run:	No

Active	Chan.	Analyte		Span	Span	Offset
		Name	Units	Units	Volts	Volts
Yes	1	O2	%	10	1	0
Yes	7	CO2	%	25	10	0

} Ranges

Measurement System Preparation Table

[illegible]

Gas Name	Zero Reference Cylinder			Low Reference Cylinder		
	No.	Conc	ID Number	No.	Conc	ID Number
O2	1	0	CC 115553	99	1	
CO2	1	0	CC 115553	99	1	

Gas Name	Mid No.	Reference Conc	Cylinder ID Number	High No.	Reference Conc	Cylinder ID Number
O2	2	5.02	SA 10790	3	7.92	CC 74709
CO2	2	14.02	SA 10790	3	20.78	CC 74709

Seq Calibration Error Test Sequence

Num	O2	CO2
-----	----	-----

1 Zero Zero

2 High High

3 Mid Mid

4

5

6

7

8

9

10

11

12

13

14

15

Seq Calibration Error Valve Sequence

Num	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16
-----	---	---	---	---	---	---	---	---	---	----	----	----	----	----	----	----

1 X

2 X

3 X

4

5

6

7

8

9

10

11

12

13

14

15

Calibration Error Test, Run 1 STRATA Version 1.0

	O2	CO2
	%	%
10-22-1999 12:49:52	-0.012	-0.061
10-22-1999 12:50:52	1.077	3.250
10-22-1999 12:51:53	7.913	20.936
10-22-1999 12:52:53	7.256	18.886

Cal + Linearity

Calibration Error Test at Run 1

Operator: Erick Mirabella
 Plant Name: Stockton CoGen (Air Products)
 Location: CFBC (Baghouse Inlet & Stack)

Reference Cylinder Numbers

	Zero	Low-range	Mid-range	High-range
O2	CC 115553		SA 10790	CC 74709
CO2	CC 115553		SA 10790	CC 74709

Span Gases

Date/Time 10-22-1999 12:53:43

PASSED

Analyte	O2	CO2
Units	%	%
Zero Ref Cyl	0.000	0.000
Zero Avg	0.002	0.000
Zero Error%	0.0%	0.0%

Zero

Low Ref Cyl		
Low Avg		
Low Error%		

Mid Ref Cyl	5.020	14.020
Mid Avg	5.037	14.045
Mid Error%	0.2%	0.1%

As Found

High Ref Cyl	7.920	20.780
High Avg	7.919	20.780
High Error%	0.0%	0.0%

Calibration

Calibration Error Test End

CERTIFICATE OF ANALYSIS / EPA PROTOCOL GAS

CUSTOMER AVOGADRO GROUP

P.O NUMBER

REFERENCE STANDARD

COMPONENT	NIST SRM NO.	CYLINDER NO.	CONCENTRATION
OXYGEN GMIS	vs. 2658a	SA 11101	5.02%
CARBON DIOXIDE GMIS	vs. 2745	282185	14.01 %

ANALYZER READINGS

R=REFERENCE STANDARD

Z=ZERO GAS

C=GAS CANDIDATE

1. COMPONENT	OXYGEN	GMIS	ANALYZER MAKE-MODEL-S/N	Siemens Oxymat 5E	S/N A12-839
ANALYTICAL PRINCIPLE			Paramagnetic	LAST CALIBRATION DATE 09/21/98	
FIRST ANALYSIS DATE			10/06/98	SECOND ANALYSIS DATE	
Z	0.00	R 5.02	C 5.02	CONC. 5.02	Z R C CONC.
R	5.02	Z 0.00	C 5.01	CONC. 5.01	R Z C CONC.
Z	0.00	C 5.02	R 5.02	CONC. 5.02	Z C R CONC.
U/M %		MEAN TEST ASSAY 5.02 %		U/M % MEAN TEST ASSAY	
2. COMPONENT	CARBON DIOXIDE	GMIS	ANALYZER MAKE-MODEL-S/N	Siemens Ultramat 5E	S/N A12-730
ANALYTICAL PRINCIPLE			NDIR	LAST CALIBRATION DATE 09/21/98	
FIRST ANALYSIS DATE			10/06/98	SECOND ANALYSIS DATE	
Z	0.00	R 14.06	C 14.06	CONC. 14.01	Z R C CONC.
R	14.06	Z 0.00	C 14.08	CONC. 14.03	R Z C CONC.
Z	0.00	C 14.08	R 14.08	CONC. 14.01	Z C R CONC.
U/M %		MEAN TEST ASSAY 14.02 %		U/M % MEAN TEST ASSAY	

Values not valid below 150 psig

THIS CYLINDER NO. SA 10790

HAS BEEN CERTIFIED ACCORDING TO SECTION

EPA-600/R97/121

OF TRACEABILITY PROTOCOL NO.

Rev. 9/97

PROCEDURE G1

CERTIFIED ACCURACY ± 1 % NIST TRACEABLE

CYLINDER PRESSURE 2000 PSIG

CERTIFICATION DATE 10/06/98

EXPIRATION DATE 10/06/01 TERM 36 MONTHS

CERTIFIED CONCENTRATION

OXYGEN	5.02 %
CARBON DIOXIDE	14.02 %
NITROGEN	BALANCE

ANALYZED BY

STEVE GUTIERREZ

CERTIFIED BY

JOSEPH CHARLES

IMPORTANT

Information contained herein has been prepared at your request by qualified experts within Praxair Distribution, Inc. While we believe that the information is accurate within the limits of the analytical methods employed and is complete to the extent of the specific analyses performed, we make no warranty or representation as to the suitability of the use of the information for any particular purpose. The information is offered with the understanding that any use of the information is at the sole discretion and risk of the user. In no event shall the liability of Praxair Distribution, Inc., arising out of the use of the information contained herein exceed the fee established for providing such information.

CERTIFICATE OF ANALYSIS / EPA PROTOCOL GAS

CUSTOMER AVAGADRO

P.O NUMBER

REFERENCE STANDARD

COMPONENT	NIST SRM NO.	CYLINDER NO.	CONCENTRATION
OXYGEN GMIS	vs. 2658a	SA 9818	10.02%
CARBON DIOXIDE GMIS	vs. 2745	282185	14.01 %

ANALYZER READINGS

R=REFERENCE STANDARD

Z=ZERO GAS

C=GAS CANDIDATE

1. COMPONENT	OXYGEN	GMIS	ANALYZER MAKE-MODEL-S/N	Siemens Oxymat 5E S/N A12-839
ANALYTICAL PRINCIPLE	Paramagnetic			LAST CALIBRATION DATE 03/13/98
FIRST ANALYSIS DATE	04/08/98			SECOND ANALYSIS DATE
Z 0.00	R 10.02	C 7.92	CONC. 7.92	Z R C CONC.
R 10.02	Z 0.00	C 7.92	CONC. 7.92	R Z C CONC.
Z 0.00	C 7.92	R 10.02	CONC. 7.92	Z C R CONC.
U/M %	MEAN TEST ASSAY 7.92 %	U/M %	MEAN TEST ASSAY	

2. COMPONENT	CARBON DIOXIDE	GMIS	ANALYZER MAKE-MODEL-S/N	Siemens Ultramat 5E S/N A12-730
ANALYTICAL PRINCIPLE	NDIR			LAST CALIBRATION DATE 03/13/98
FIRST ANALYSIS DATE	04/08/98			SECOND ANALYSIS DATE
Z 0.00	R 14.00	C 14.40	CONC. 14.41	Z R C CONC.
R 14.00	Z 0.00	C 14.42	CONC. 14.43	R Z C CONC.
Z 0.00	C 14.41	R 13.98	CONC. 14.44	Z C R CONC.
U/M %	MEAN TEST ASSAY 14.43 %	U/M %	MEAN TEST ASSAY	

Values not valid below 150 psig; STEC GAS DIVIDER SDG-710
70% SPLIT, POINT CORRECTION FACTOR 0.694328 ASSAY=20.78% CARBON DIOXIDE

THIS CYLINDER NO.	CC 74709	CERTIFIED CONCENTRATION
HAS BEEN CERTIFIED ACCORDING TO SECTION	EPA-600/R97/121	OXYGEN 7.92 %
OF TRACEABILITY PROTOCOL NO.	Rev. 9/97	CARBON DIOXIDE 20.78 %
PROCEDURE	G2	NITROGEN BALANCE
CERTIFIED ACCURACY	± 2 % NIST TRACEABLE	
CYLINDER PRESSURE	2000 PSIG	
CERTIFICATION DATE	04/08/98	
EXPIRATION DATE	04/08/01 TERM 36 MONTHS	

ANALYZED BY

JOSEPH CHARLES

CERTIFIED BY

KWAN YOUNG

DRY GAS METER CALIBRATION CHECK

Calibration Date:

7/8/99

Calibrated by:

Peter Gates

Meter #

N-2

Serial #

81083

(Q)	Flow Rate (scfm)	(Pb) Barometric Pressure (in Hg)	(DH) Orifice Pressure Drop	(DP) Wet Test Meter Drop	(Vds) Wet Test Meter Yd	Wet Test Meter Volume (ft ³)			Dry Gas Meter Volume (ft ³)			Wet Test Meter Temperature (°F)			Dry Gas Meter Temperature (°F)			(dt) Elapsed Time (hr:min:sec)	(Yd) Dry Gas Meter Yd	(DH@) Orifice DH @ 0.75 cfm
						Initial	Final	Net	Initial	Final	Net	In	Out	Avg	In	Out	Avg			
0.401		29.70	0.50	-0.50	1.0	0	5.296	5.296	653.085	658.343	5.258	72.0	72.0	72.0	80.5	78.5	79.5	00:13:00	1.0189	1.8559
0.876		29.70	1.50	-1.50	1.0	0	5.414	5.414	667.853	673.062	5.408	71.3	71.3	65.0	86.0	81.5	83.8	00:08:00	1.0290	1.7708
0.860		29.70	2.50	-2.00	1.0	0	5.236	5.236	680.257	685.540	5.283	71.7	71.7	71.7	90.0	83.0	86.5	00:06:00	1.0075	1.8112

Equations:

$$Q = (17.836 \times Yds \times Vds \times Pb) / (dt \times (Tds + 460))$$

$$Yd = Yds \times (Vds/Vd) \times \{(Tds + 460)/(Tds + 460)\} \times \{(Pb + DP/13.6)/(Pb + DH/13.6)\}$$

$$DH@ = \{0.0317 \times DH / (Pb \times (Td + 460))\} \times \{(Tds + 460) \times t / (Vds \times Yds)\}^2$$

Calibrated Value, Yd =

1.018

Calibrated Value, DH@ =

1.756

The Avogadro Group

POST-TEST DRY GAS METER CALIBRATION DATA

CALIBRATED BY Peter Gates
 DATE 10-25-99
 FIELD METER ID N-2
 BAROMETRIC PRESS 29.93

TEST METER ID 1039745
 TEST METER Y (Y) 1.0005
 TEST METER LAST CAL 1-16-99

TEST PROGRAM PRECEDING CALIBRATION CHECK Stockton Cogen
 AVERAGE ΔH FROM TEST RUNS 0.40
 MAXIMUM VACUUM FROM TEST RUNS 7.0
 LEAK CHECK VAC 7.0 INIT. 0.006 CFM

FIELD METER								TEST METER					
			TEMPERATURE								TEMPERATURE		
ΔH	FINAL VOLUME	INITIAL VOLUME	VOLUME (V _{in})	INLET (t)	OUTLET (t)	VACUUM	TIME (0)	FINAL VOLUME	INITIAL VOLUME	VOLUME (V _m)	INLET	OUTLET	METER PRESS.
0.40	329.455	324.188	5.267	69/76	69/74	7.0	15:00	423.645	418.311	5.334	69/70	69/69	- .35
0.40	334.734	329.455	5.279	83/88	76/79	7.0	15:00	428.917	423.645	5.272	71/72	69/70	- .35
0.40	340.021	334.734	5.287	94/100	82/83	7.0	15:00	434.117	428.917	5.200	74/75	70/71	- .35

THE AVOGADRO GROUP
EPA METHOD 5
Post-Test Meter Cal.
Stockton Cogen

FIELD METER

Meter #: N-2
 Meter Yd: 1.0180
 Bar. Press.: 29.93

TEST METER

TM ID #: 1039745
 TM Yd: 1.0005
 Last Cal : 1/16/99

CALIBRATION DATA

Date: 10/25/99
 Cal By: Peter Gates
 Yd <=3% of Average

FIELD METER DATA					TEST METER DATA								
DELTA H	TIME	INITIAL VOLUME	FINAL VOLUME	VOLUME ft ³	TEMP °F	INLET VOLUME	OUTLET VOLUME	INITIAL VOLUME	FINAL VOLUME	VOLUME ft ³	TEMP °F	INLET	OUTLET
"H ₂ O													
Average	15.00	324.188	329.455	5.267	69	69	69	418.311	423.645	5.334	69	69	69
0.40					76	76	74				70	70	69
Average	15.00	329.455	334.734	5.279	83	76	76	423.645	428.917	5.272	71	69	69
0.40					88	79	79				72	70	70
Average	15.00	334.734	340.021	5.287	94	82	82	428.917	434.117	5.200	74	70	70
0.40					100	83	83				75	71	71

METER Yd:

1.0175
 1.0189
 1.0149

DELTA H:

0.000
 0.002
 -0.002

AVERAGE 1.0171 AVERAG 1.790

% DIFFERENCE FROM LAST Yd= 0.088

DRY GAS METER FULL CALIBRATION (6 mo.)

Calibration Date: 6/18/99

Meter # N - 3
Serial # 28453

Calibrated by: Peter Gates

(Q) Flow Rate (scfm)	(Pb) Barometric Pressure (in Hg)	(DH) Orifice Pressure Drop	(DP) Wet Test Meter DP	(Yds) Wet Test Meter Yd	(Vds) Wet Test Meter Volume (ft ³)			(Vd) Dry Gas Meter Volume (ft ³)			(Tds) Wet Test Meter Temperature (°F)			(Td) Dry Gas Meter Temperature (°F)			(dt) Elapsed Time (hr:min:sec)	(Yd) Dry Gas Meter Yd	(DH@) Orifice DH @ 0.75 cfm
					Initial	Final	Net	Initial	Final	Net	In	Out	Avg	In	Out	Avg			
0.391	29.70	0.50	-5.10	1.0	0	5.000	5.000	118.717	123.746	5.029	73.0	73.0	73.0	82.0	74.0	78.0	00:12:35	0.9897	1.7837
0.674	29.70	1.50	-1.50	1.0	0	5.005	5.005	124.308	129.373	5.065	73.0	73.0	73.0	85.0	76.0	80.5	00:07:18	0.9947	1.7890
0.870	29.70	2.50	-2.40	1.0	0	5.000	5.000	130.948	135.953	5.005	73.0	73.0	73.0	88.0	76.0	82.0	00:05:39	1.0036	1.7848

Equations:

$$Q = (17.636 \times Yds \times Vds \times Pb) / (dt \times (Tds + 460))$$

$$Yd = Yds \times (Vds/Vd) \times ((Td+460)/(Tds+460)) \times ((Pb + DP/13.6)/(Pb+DH/13.6))$$

$$DH@ = \{0.0317 \times DH / [Pb \times (Td+460)]\} \times \{ (Tds+460) \times t / (Vds \times Yds) \}^2$$

Calibrated Value, Yd = 0.996

Calibrated Value, DH@ = 1.786

The Avogadro Group

POST-TEST DRY GAS METER CALIBRATION DATA

CALIBRATED BY Peter Gates
 DATE 10-25-99
 FIELD METER ID N-3
 BAROMETRIC PRESS 29.93

TEST METER ID 1039745
 TEST METER Y (Y) 1.0005
 TEST METER LAST CAL 1-16-99

TEST PROGRAM PRECEDING CALIBRATION CHECK Stockton Cogen
 AVERAGE ΔH FROM TEST RUNS 0.90
 MAXIMUM VACUUM FROM TEST RUNS 4.0
 LEAK CHECK VAC 4.0 INIT. 0.010 CFM

FIELD METER								TEST METER					
				TEMPERATURE							TEMPERATURE		
ΔH	FINAL VOLUME	INITIAL VOLUME	VOLUME (V _{eq})	INLET (t)	OUTLET (t)	VACUUM	TIME (0)	FINAL VOLUME	INITIAL VOLUME	VOLUME (V _m)	INLET	OUTLET	METER PRESS.
0.90	2420.40	234.682	7.358	68/74	67/70	4.0	15:00	819.409	812.012	7.397	/	/	-.80
0.90	249.441	242.040	7.401	75/82	71/76	4.0	15:00	826.786	819.409	7.377	/	/	-.80
0.90	256.945	249.441	7.504	83/92	78/83	4.0	15:00	834.203	826.786	7.417	/	/	-.80

THE AVOGADRO GROUP
EPA METHOD 5
Post-Test Meter Cal.
Stockton Cogen

FIELD METER

Meter #: N-3
 Meter Yd: 0.9960
 Bar. Press.: 29.93

TEST METER

TM ID #: 1039745
 TM Yd: 1.0005
 Last Cal : ~~6/99~~ 1/16/11

CALIBRATION DATA

Date: 10/25/99
 Cal By: Peter Gates
 Yd <= 3% of Average

FIELD METER DATA					TEST METER DATA								
DELTA H	TIME	INITIAL VOLUME	FINAL VOLUME	VOLUME ft ³	VOLUME TEMP 'F	INLET VOLUME	OUTLET VOLUME	INITIAL VOLUME	FINAL VOLUME	VOLUME ft ³	TEMP 'F	INLET	OUTLET
"H ₂ O													
Average	15.00	234.682	242.040	7.358	68	67		812.012	819.409	7.397		71	71
0.90					74	70						75	72
Average	15.00	242.040	249.441	7.401	75	71		819.409	826.786	7.377		77	73
0.90					82	76						79	74
Average	15.00	249.441	256.945	7.504	83	78		826.786	834.203	7.417		80	74
0.90					92	83						81	75

METER Yd:

0.9989 0.001
 0.9955 -0.002
 0.9987 0.001

DELTA H:

2.098 0.004
 2.113 0.018
 2.073 -0.022

AVERAGE 0.9977

AVERAG 2.094

DIFFERENCE FROM LAST Yd= 0.0017

% DIFFERENCE FROM LAST Yd= -0.169

EPA METHOD 2 (Outlet) PROBE ASSEMBLY AND CALIBRATIONS

Technician:

Date:

DD | EM
10/16/99

Pre-test

Probe Assembly:

Probe Assembly Number:	A-10B	Serial #	Result	Notes / Limits:
Thermocouple Numbers (stack):	T-10B _S	--	--	1-inch sheath
Thermocouple Numbers (probe):	T-10B _P	--	--	K-type
Probe Liner Number:	L-10B	--	--	K-type
Nozzle Number:	0.171	--	--	10 feet, quartz
Pitot Number:	P-10B	--	--	button-hook, quartz
				stainless steel

Pitot Alignment Specifications:

Transverse tube axis (are face openings aligned)	--	Yes	must be aligned
External tubing diameter (D _t)	-- 23/64	0.3594	between 3/16 (0.1875) in. and 3/8 (0.3750) in.
Base to opening plane distances (P _A and P _B)	-- 1/2	0.50	between 1.05 and 1.50 D _t (0.3774 - 0.5391)
Are P _A and P _B equal?	--	Yes	must be equal
Distance between pitot tube and nozzle	--	3/4	should be at least 3/4 inch
Distance between pitot tube and TC	--	3/4	between 3/4 and 1 inch
Distance between pitot tube and probe	--	S	should be at least 3 inches

Miscellaneous:

Are aerodynamic interference effects eliminated?	--	Yes	impact pressure opening plane of pitot must be below entry plane of nozzle
Has probe been inspected prior to mobilization?	--	Yes	leak checks, heating elements, TC response, probe and nozzle condition within the last 6 months
Are all calibrations current?	--	Yes	See CFR
Are calibrations within individual specifications?	--	Yes	date, call airport Stockton Airport
Barometer calibration	-- 30.07	10/19	must be verified on site - New
Nozzle calibration	-- 0.171	10/19	use 0.84 if it meets the criteria above
Pitot coefficient	--	0.84	

EPA METHOD 2

PROBE ASSEMBLY AND CALIBRATIONS

Technician: EM

Date: 10/22/99

Post Check

Probe Assembly:

Probe Assembly Number:	A-10B	Result	--	Notes / Limits:	1-inch sheath
Thermocouple Numbers (stack):	T-10Bs	Result	--		K-type
Thermocouple Numbers (probe):	T-10Bp	Result	--		K-type
Probe Liner Number:	L-10B	Result	--		10 feet, quartz
Nozzle Number:	0.171	Result	--		button-hook, quartz
Pitot Number:	P-10B	Result	--		stainless steel

Pitot Alignment Specifications:

Transverse tube axis (are face openings aligned)	--	<u>Yes</u>	must be aligned	✓
External tubing diameter (D_t)	--	<u>23/64</u>	between 3/16 (0.1875) in. and 3/8 (0.3750) in.	✓
Base to opening plane distances (P_A and P_B)	--	<u>1/2</u>	between 1.05 and 1.50 D_t	✓
Are P_A and P_B equal?	--	<u>Yes</u>	must be equal	✓
Distance between pitot tube and nozzle	--	<u>3/4</u>	should be at least 3/4 inch	✓
Distance between pitot tube and TC	--	<u>3/4</u>	between 3/4 and 1 inch	✓
Distance between pitot tube and probe	--	<u>5</u>	should be at least 3 inches	✓

Miscellaneous:

Are aerodynamic interference effects eliminated?	--	<u>Yes</u>	impact pressure opening plane of pitot must be below entry plane of nozzle	✓
Has probe been inspected prior to mobilization?	--	<u>Yes</u>	leak checks, heating elements, TC response, probe and nozzle condition	✓
Are all calibrations current?	--	<u>Yes</u>	within the last 6 months	✓
Are calibrations within individual specifications?	--	<u>Yes</u>	See CFR	✓
Barometer calibration	--	<u>10/22</u>	date, call airport	<u>29.74</u> - read <u>29.76</u>
Nozzle calibration	--	<u>10/22</u>	must be verified on site	✓
Pitot coefficient	--	<u>0.84</u>	use 0.84 if it meets the criteria above	✓

EPA METHOD 2 (Inlet) PROBE ASSEMBLY AND CALIBRATIONS

Technician:

Date:

DD/EM
10/16/99

Pre-test

Probe Assembly:

Probe Assembly Number:	A-9D	Serial #	Result	Notes / Limits:
Thermocouple Numbers (stack):	T-9D _s		--	3-inch sheath
Thermocouple Numbers (probe):	T-9D _p		--	K-type
Probe Liner Number:	L-9D		--	K-type
Nozzle Number:	0.155		--	9 feet, quartz
Pitot Number:	P-9D		--	button-hook, quartz
				stainless steel

Pitot Alignment Specifications:

Transverse tube axis (are face openings aligned)	--	Yes	must be aligned
External tubing diameter (D _t)	-- 11.32	0.3438	between 3/16 (0.1875) in. and 3/8 (0.3750) in.
Base to opening plane distances (P _A and P _B)	-- 11.2	0.50	between 1.05 and 1.50 D _t (0.3610 - 0.5157)
Are P _A and P _B equal?	--	Yes	must be equal
Distance between pitot tube and nozzle	-- 0.81	11.16	should be at least 3/4 inch (0.75)
Distance between pitot tube and TC	--	3.4	between 3/4 and 1 inch
Distance between pitot tube and probe	--	5	should be at least 3 inches

Miscellaneous:

Are aerodynamic interference effects eliminated?	--	Yes	impact pressure opening plane of pitot must be below entry plane of nozzle
Has probe been inspected prior to mobilization?	--	Yes	leak checks, heating elements, TC response, probe and nozzle condition within the last 6 months
Are all calibrations current?	--	Yes	See CFR
Are calibrations within individual specifications?	--	Yes	date, call airport Stockton Airport
Barometer calibration	-- 30.07	10.19	must be verified on site - New
Nozzle calibration	-- 0.155	10.19	use 0.84 if it meets the criteria above
Pitot coefficient	--	0.84	

EPA METHOD 2

PROBE ASSEMBLY AND CALIBRATIONS

Technician:

EM

Date:

10/22/99

Probe Assembly:

Probe Assembly Number:	Serial #	Result	Notes / Limits:
Thermocouple Numbers (stack):	A-9D	--	3-inch sheath
Thermocouple Numbers (probe):	T-9D _s	--	K-type
Probe Liner Number:	T-9D _p	--	K-type
Nozzle Number:	L-9D	--	9 feet, quartz
Pitot Number:	0.155	--	button-hook, quartz
	P-9D	--	stainless steel

Pitot Alignment Specifications:

Transverse tube axis (are face openings aligned)	--	Yes	must be aligned ✓
External tubing diameter (D _t)	--	11/32	between 3/16 (0.1875) in. and 3/8 (0.3750) in. ✓
Base to opening plane distances (P _A and P _B)	--	1/2	between 1.05 and 1.50 D _t ✓
Are P _A and P _B equal?	--	Yes	must be equal ✓
Distance between pitot tube and nozzle	--	11/16	should be at least 3/4 inch ✓
Distance between pitot tube and TC	--	3/4	between 3/4 and 1 inch ✓
Distance between pitot tube and probe	--	5	should be at least 3 inches ✓

Miscellaneous:

Are aerodynamic interference effects eliminated?	--	Yes	impact pressure opening plane of pitot must be below entry plane of nozzle ✓
Has probe been inspected prior to mobilization?	--	Yes	leak checks, heating elements, TC response, probe and nozzle condition ✓
Are all calibrations current?	--	Yes	within the last 6 months ✓
Are calibrations within individual specifications?	--	Yes	See CFR ✓
Barometer calibration	--	10/22	date, call airport 29.74 - read 29.76 ✓
Nozzle calibration	--	10/22	must be verified on site ✓
Pitot coefficient	--	0.84	use 0.84 if it meets the criteria above ✓

Post Checks

Probe Assembly A-10B

STACK TEMPERATURE SENSOR CALIBRATION DATA FORM

CLIENT: Avogadro Group

THERMOCOUPLE NO.: TYPE-K T-10Bs

DATE: 10/14/99

AMBIENT TEMP., °F: 76°F

OPERATOR: PG

BAROMETRIC PRESS. (in. Hg): 29.89

CALIBRATOR: EM

REF. (MERCURY-IN-GLASS): 73°F

NAME: Erick Mirabella / Peter Gates

SERIAL #: RT #'s 1, 2, 3 (Ref.)

REFERENCE POINT NUMBER*	SOURCE ^b (SPECIFY)	REFERENCE THERMOMETER TEMPERATURE, °F	THERMOCOUPLE POTENTIOMETER TEMPERATURE, °F	TEMPERATURE DIFFERENCE, °C %
COLD	T-10B ICE WATER		34°F	0.08 ✓
	T-10Bs		36°F	0.49 ✓
	T-10Bp	0.9°C = 33.6°F	37°F	0.69 ✓
MEDIUM	BOILING WATER			
	T-10Bs		215°F	0.99 ✓
	T-10Bp	98°C = 208.4°F	213°F	0.69 ✓
HOT	HOT OIL			
	T-10Bs		372°F	0.85 ✓
	T-10Bp	185°C = 365°F	369°F	0.48 ✓
Medium	Oven Temp			
	T-06	137°C = 278.6	273°F	0.76 ✓

*EVERY 100°F FOR EACH REFERENCE POINT.

^bTYPE OF CALIBRATION SYSTEM USED.

$$^{\circ} \left[\frac{(\text{REF. TEMP., } ^{\circ}\text{F} + 460) - (\text{TEST THERMOM. TEMP., } ^{\circ}\text{F} + 460)}{\text{REF. TEMP., } ^{\circ}\text{F} + 460} \right] 100 \leq 1.5\%$$



Probe Assembly A-90

STACK TEMPERATURE SENSOR CALIBRATION DATA FORM

CLIENT: Avogadro
 DATE: 10/14/99
 OPERATOR: PG
 CALIBRATOR: EM
 NAME: Erick Mirabella / Peter Gates

THERMOCOUPLE NO.: TYPE-K
 AMBIENT TEMP., °F: 79°F
 BAROMETRIC PRESS. (in. Hg): 29.85
 REF. (MERCURY-IN-GLASS): 75°F
 SERIAL #: Ref. RT#s 1,2,3

REFERENCE POINT NUMBER*	SOURCE* (SPECIFY)	REFERENCE THERMOMETER TEMPERATURE, °F	THERMOCOUPLE POTENTIOMETER TEMPERATURE, °F	TEMPERATURE DIFFERENCE, ° %
COLD	T-V2 ICE WATER T-9Ds	1°C = 33.8°F	33°F	0.16 ✓
			35°F	0.24 ✓
			34°F	0.04 ✓
MEDIUM	T-9DP BOILING WATER T-9Ds	99°C = 210.2°F	212 °F	0.27 ✓
			216 °F	0.87 ✓
HOT	T-9DP HOT OIL T-9Ds	181°C = 357.8°F	363°F	0.64 ✓
			368°F	1.25 ✓
Medium	Oven Temp T-04	134°C = 273.2°F	269°F	0.57 ✓

*EVERY 100°F FOR EACH REFERENCE POINT.

*TYPE OF CALIBRATION SYSTEM USED.

$$\left[\frac{(\text{REF. TEMP., } ^\circ\text{F} + 460) - (\text{TEST THERMOM. TEMP., } ^\circ\text{F} + 460)}{\text{REF. TEMP., } ^\circ\text{F} + 460} \right] 100 \leq 1.5\%$$

VELOCITY TRAVERSE DATA

CLIENT/LOCATION: Stockton Cogen

DATE: 10-19-99

SAMPLE LOCATION: Boiler Outlet

DATA TAKEN BY:

UNIT NO.: Boilers

TEST DESCRIPTION: Prelim traverse

TEST NO.: ✓ Vel + Cyclonic Flow

BARO. PRESS. (in. Hg): 29.95

PITOT TUBE COEFFICIENT 0.84 C_p

ABS. STATIC PRESS. IN STACK (in. Hg) -0.84 P_s

$$V_s = 2.90 \text{ Cp} \sqrt{\Delta P} T_s \sqrt{\frac{29.92}{P_s} \times \frac{28.95}{\text{MW}}}$$

[illegible]

No Cyclonic Flow

$$\Delta P = 1.2587$$

Temp. = 280.2

- ~ 1790

- ~ 10%



From last year

DCBA

N ↑

~~Initial Transverse~~
Cyclonic Flow

SAMPLE TRAIN TEST DATA

UNIT Bayhuse Inhibit TEST NO. Int Transverse METHOD 2 - Prodim, PAGE 1 OF 1
TEST CONDITION Full Load AMB. TEMP., °F _____ PROJECT # 99017
METER VOL. (START/END) _____ DATE 10-19-99
CLIENT Air Products
SAMPLE LOCATION Stacks C/A
OPERATOR/ASSISTANT KC/JP

PRE-TEST DATA:

Barometric Press., in. Hg.
Assumed Moisture
Assumed Molecular Wt.
Assumed ΔP
Assumed ΔH
Stack Diameter, in.
Sample Time: Total
per point
Total of Traverse Points

$$\Delta H = \frac{\Delta P}{x}$$

EQUIPMENT INFO:

Meter No.	
Meter, Yd.	
CFM @ $\Delta H = 1.0$	
Pilot ID, Cp	
O_2/CO_2 Method	
Teflon Connecting	
Line (Y/N)	
Probe:	Mat'l
Length	
Nozzle:	Mat'l
Diam.	
Filter:	No.
	Mat'l

POST TEST INFO:

Filter Appearance
Impinger Appearance
Silica Gel Spent (Y/N)

151

[illegible]

SAMPLE TRAIN LEAK CHECK:

	CFM	Vac.	Pinot	Init.
Pre-Test	_____	_____	_____	_____
Post-Test	_____	_____	_____	_____

PRE-TEST CALIBRATION CHECK:

	Time	ΔH	Meter Reading	Meter Temp.
			In	Out
Init.	_____	_____	_____	_____
Final	_____	_____	_____	_____

[illegible]

SAMPLE TRAIN TEST DATA

CLIENT <u>Air Products</u>	UNIT <u>Boiler</u>	TEST NO. <u>1-40-Inter</u>	METHOD <u>OF 1</u>
SAMPLE LOCATION <u>Stockton</u>	TEST CONDITION <u>FULL</u>	AMB. TEMP., °F <u>70.5</u>	PROJECT # <u>9057</u>
OPERATOR/ASSISTANT <u>PKC</u>	METER VOL. (START/END) <u>10.9.99</u>	DATE <u>10.9.99</u>	

PRE-TEST DATA:			
Barometric Press., in. Hg.	29.95		
Assumed Slack Press.			
Assumed Moisture			
Assumed Molecular Wt.			
Assumed ΔP			
Assumed ΔH			
Slack Diameter, in.	84" X		
Sample Time: Total	10		
per point	10		
Total of Traverse Points	1		
ΔH = 12		x ΔP	

EQUIPMENT INFO:			
Meter No.			
Meter, Yd.			
CFM @ ΔH = 1.0			
Pilot ID, Cp			
O ₂ /CO ₂ Method			
Tellon Connecting			
Line (Y/N)			
Probe:			
Mat'l			
Length			
Nozzle:			
Mat'l			
Diam.			
Filter:			
No.			
Mat'l			

SAMPLE TRAIN LEAK CHECK:			
CFM	Vac.	Pilot	Init.
Pre-Test	0.002	10	
Post-Test	0.004	24	

PRE-TEST CALIBRATION CHECK:			
Time	ΔH	Meter Reading	Meter Temp.
Init.			In
Final			Out

POST TEST INFO:			
Filter Appearance			
Impinger Appearance			
Silica Gel Spent (Y/N)			

[illegible]

APPENDIX C

DATA SHEETS

Appendix C.1

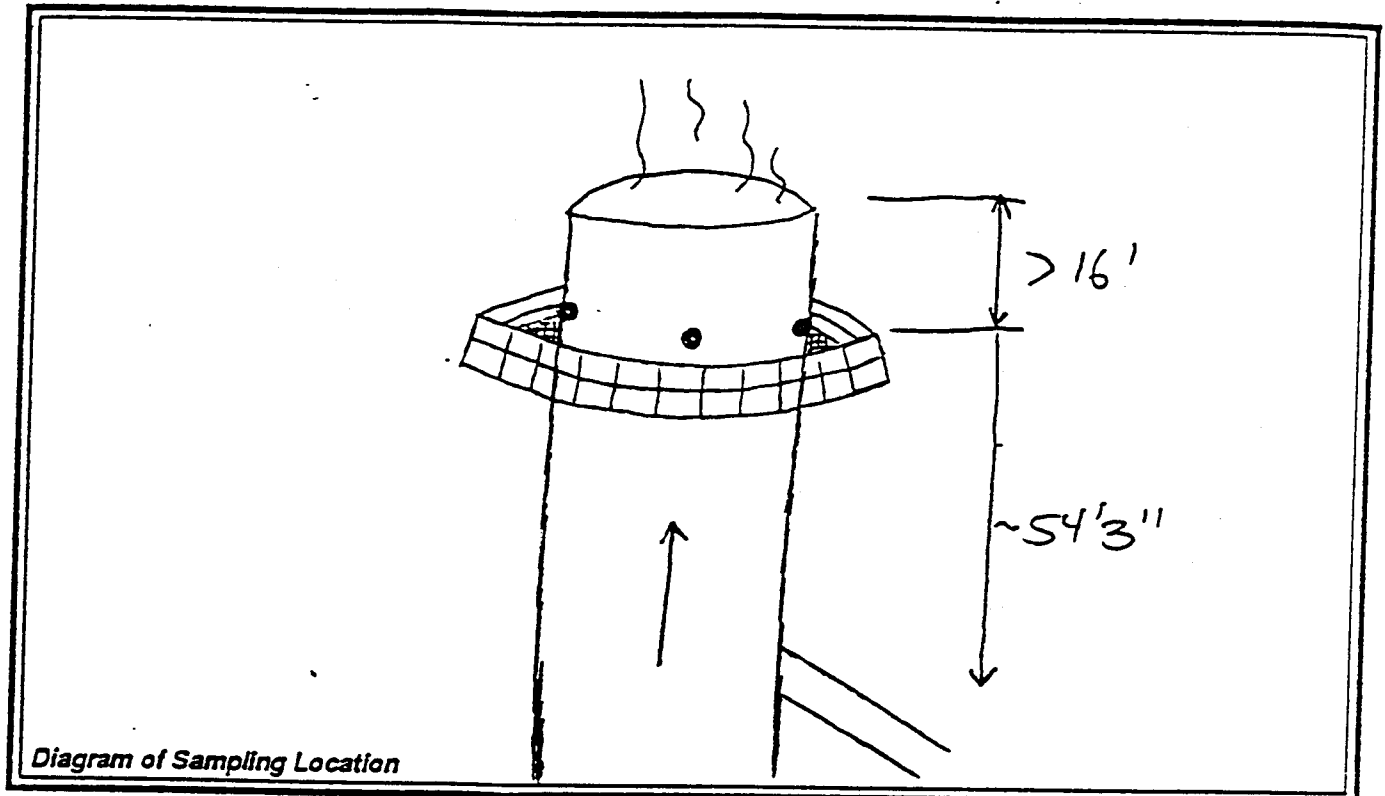
Sampling Locations

SAMPLING POINT LOCATION DATA - EPA METHOD 1

PLANT: Air Products / Stockton DATA BY: EM

DATE: 10/20/99

TEST LOCATION: CFBC Boiler Stack (Outlet)



UPSTREAM DIST./DIA.: 54'3" - 6.8

DOWNSTREAM DIST./DIA.: 716' / 72

COUPLING LENGTH: 12.5

NO. OF SAMPLING PTS.: 16

STACK DIMENSION: 8.0' (96")

STACK AREA, FT²: 50.265

SAMPLE POINT	% OF DIAMETER	IN. FROM NEAR WALL	IN. FROM NOZZLE*
1	3.2	3.1	15.6
2	10.5	10.1	22.6
3	19.4	18.6	31.1
4	32.3	31.0	43.5
5	67.7	65.0	77.0
6	80.6	77.4	89.9
7	89.5	86.0	98.5
8	96.8	92.9	105.4
1	16.7	16.0	28.5
2	50.0	48.0	60.5
3	83.3	80.0	92.5

*INCHES FROM WALL PLUS
COUPLING LENGTH

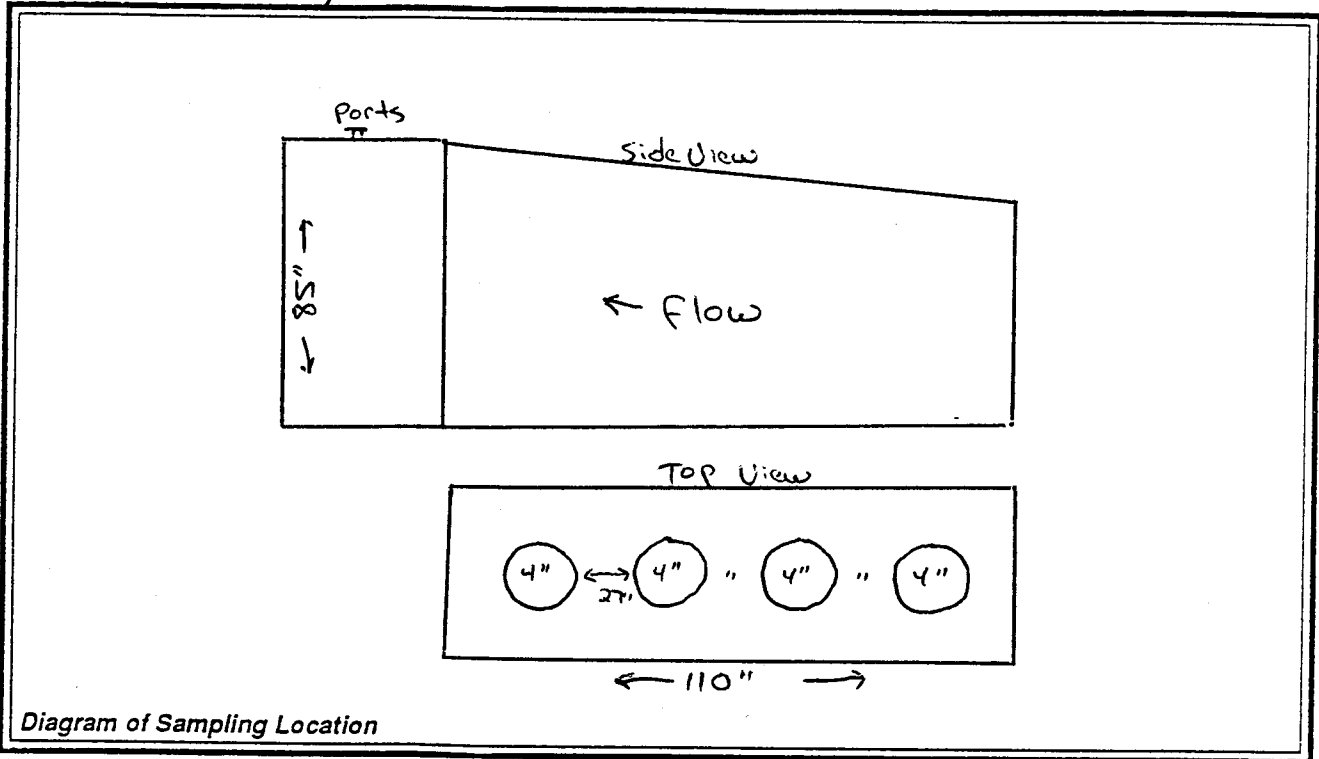
SAMPLING POINT LOCATION DATA - EPA METHOD 1

PLANT: Stockton Cofen

DATA BY: EM

DATE: 10/20/99

TEST LOCATION: Baghouse Inlet



Does not meet EPA 1 Criteria.

UPSTREAM DIST./DIA.: ~ 3'

DOWNSTREAM DIST./DIA.: ~ 3'

COUPLING LENGTH: 12" + 7" ext = 19"

NO. OF SAMPLING PTS.: 6 x 4 = 24

STACK DIMENSION: 85" x 110"

STACK AREA, FT²: 64.93

SAMPLE POINT	% OF DIAMETER	IN. FROM NEAR WALL	IN. FROM NOZZLE*
1	91.7	77.9"	96.9"
2	75.0	63.8"	82.8"
3	58.3	49.6"	68.6"
4	41.7	35.4"	54.4"
5	25.0	21.3"	40.3"
6	8.3	7.1"	26.1"

*INCHES FROM WALL PLUS COUPLING LENGTH

Appendix C.2

Plant Process and CEMS Data

YEAR	MONTH	99	KPH										PPH	LIME	TEMP	KPH	MW	Coke	COKE	PPH	PPH	F	Coke	
			BAGH				COAL	FEED	CWF1	CWF2	CWF3	CWF4												CWF5
			BAGH	DP	IN	TEMP																		
DAY	20	10	BAGH	270	8.55	59.6	7280	7248	9809	9746	7655	4903	304	516	58.4	30.0	14644	41739	14644					
HOUR	MIN	23	298	7.62	59.8	7103	7107	9535	9560	7371	5166	304	519	58.9	32.0	15716	40676	15716						
		13	298	8.81	60.3	7094	7133	9632	9587	7380	5031	305	522	59.7	32.0	15780	40826	15780						
		13	299	8.83	60.0	7059	7266	9614	9578	7415	5008	306	524	60.1	32.0	15773	40933	15773						
		14	300	8.47	59.9	6908	6956	9376	9321	7167	4968	306	525	60.0	33.8	16663	39728	16663						
		14	301	7.59	59.5	6855	6841	9296	9286	7300	4995	307	528	60.1	34.0	16657	39578	16657						
		14	300	7.96	58.2	6810	6823	9217	9259	7282	4873	307	527	59.7	33.9	16562	39392	16562						
		14	300	9.03	59.5	6713	6814	9287	9348	7194	5093	307	521	59.3	33.7	16562	39356	16562						
		14	299	8.52	59.9	6864	6947	9367	9401	7069	5035	307	522	59.9	33.9	16657	39648	16657						
		14	301	8.53	60.0	6677	6637	8987	9153	6972	5086	307	527	60.4	35.9	17508	38426	17508						
		15	302	7.69	59.0	6296	6282	8766	8737	6661	5429	308	528	60.1	37.9	18449	36743	18449						
		15	302	9.29	59.5	6349	6238	8775	8595	6431	5481	308	526	59.9	38.2	18404	36389	18404						
		15	301	8.63	59.0	6243	6229	8748	8764	5508	5616	308	523	59.6	38.1	18404	35493	18404						
		15	301	8.34	59.1	6216	6318	8775	8772	6626	5434	308	522	59.7	37.8	18455	36708	18455						
		15	302	7.55	59.6	6349	6318	8792	8817	6715	5646	309	528	60.0	38.0	18500	36991	18500						
		15	302	7.90	59.5	6367	6362	8792	8799	6661	5413	309	526	59.9	37.9	18505	36982	18505						
		16	303	8.05	59.0	5764	5875	8306	8250	6165	5566	309	534	61.0	41.9	20320	34360	20320						
		16	302	8.56	58.2	5649	5733	8165	8188	5961	5469	309	530	60.7	42.1	20094	33696	20094						
		16	302	8.19	57.7	5764	5742	8218	8197	6156	5399	309	520	59.5	41.5	19666	34077	19666						
		16	301	7.53	58.3	5649	5751	8174	8250	5047	5351	309	514	58.7	41.9	19796	32871	19796						
		16	301	8.11	58.0	5401	5458	7953	7870	5766	5596	308	523	59.8	44.6	21187	32447	21187						
Average			299.2	8.27	59.21	6448	6480	8933	8928	6690	5265	307.5	524.0	59.78	36.72	17824	37479	17824						

YEAR	MONTH	DAY	HOUR	MIN	KPH			PPH	LIME	TEMP	MN	KPH	MW	GEN	Coke	COKE	PPH	PPH	F	Coke	
					BAG	H	COAL														STM
					DP	BAG	H	COAL	FEED	CWF1	CWF2	CWF3	CWF4	CWF5	FEED						
99	10	21	10	13	8.26	283		61.7	6128	6159	8960	8941	6972	8120	290	510	57.9	39.7	20184	37159	20184
			10	23	8.84	284		61.8	5418	5467	8209	8242	6165	7825	290	521	60.0	45.0	22962	33501	22962
			10	33	8.49	285		61.8	5179	5157	8006	7994	5881	8115	291	526	61.6	48.0	24370	32216	24370
			10	43	8.78	289		60.5	4984	4998	7882	7861	5748	8013	292	522	61.1	47.9	23728	31472	23728
			10	53	7.86	297		58.6	4798	4820	7635	7684	5508	7720	295	521	60.3	48.0	23075	30445	23075
			11	3	8.55	288		58.3	4505	4493	7370	7339	5304	8198	298	497	57.8	49.9	23818	29010	23818
			11	13	8.22	284		59.3	4638	4652	7467	7471	5357	8631	297	501	58.3	50.0	24314	29586	24314
			11	23	8.40	283		59.7	4567	4537	7414	7365	5295	9360	294	508	59.5	51.2	25013	29178	25013
			11	33	7.72	284		60.3	4735	4758	7617	7551	5473	9608	294	511	59.0	50.0	24692	30135	24692
			11	43	7.93	284		60.7	4798	4838	7600	7613	5499	9773	293	517	59.4	49.9	24838	30348	24838
			11	53	8.94	285		60.6	4780	4803	7573	7595	5526	9685	294	522	59.5	50.0	24861	30277	24861
			12	3	8.42	285		60.9	4824	4812	7617	7595	5499	9976	294	523	59.0	50.1	25013	30348	25013
			12	13	8.48	286		61.3	4842	4883	7688	7648	5597	9162	294	526	60.7	50.1	25092	30658	25092
			12	23	7.96	288		60.5	4673	4661	7476	7498	5393	5857	295	527	61.3	50.9	25339	29701	25339
			12	33	8.81	288		59.5	4549	4528	7343	7383	5242	8145	296	519	60.4	50.9	24815	29045	24815
			12	43	8.23	287		58.8	4469	4510	7379	7294	5216	8195	296	509	59.2	51.0	24539	28868	24539
			12	53	8.16	286		58.8	4487	4493	7317	7330	5189	7542	296	504	58.6	51.0	24669	28815	24669
			13	3	7.60	286		59.2	4425	4448	7246	7223	5100	7893	295	508	59.6	52.1	25261	28443	25261
			13	13	7.87	287		59.2	4416	4395	7264	7241	5118	7935	295	512	60.2	52.1	25289	28434	25289
			13	23	8.83	287		59.4	4425	4448	7220	7206	5171	8511	296	512	60.4	52.0	25311	28470	25311
Average					8.32	286.4		60.04	4782	4793	7614	7604	5513	8413	294.4	514.8	59.69	49.49	24359		

YEAR MONTH	99 10	DAY	22	HOUR	MIN	BAG IN	BAG TEMP	BAG DP	COAL FEED	KPH															
										CWF1	CWF2	CWF3	CWF4	CWF5	LIME FEED	PPH	TEMP	MN FLOW	KPH	MW	GEN LOAD	Coke %	COKE KLBS/HR	PPH Coal	PPH FCoke
8	53	283	9.17	61.5	5330	5396	8200	8188	6094	8158	290	527	59.7	46.0	23176	33208	23176								
9	3	284	8.97	61.6	5347	5414	8200	8179	6111	8476	290	529	60.0	46.1	23249	33253	23249								
9	13	284	9.05	61.7	5374	5432	8236	8215	6085	8744	290	530	60.1	46.1	23277	33341	23277								
9	23	284	7.99	61.8	5401	5467	8227	8206	6085	9062	291	529	60.1	46.0	23322	33386	23322								
9	33	285	9.09	61.6	5409	5414	8209	8179	6094	9490	291	531	61.2	46.0	23249	33306	23249								
9	43	285	8.57	60.8	5276	5272	8095	8109	6041	9019	291	520	60.0	45.8	22849	32792	22849								
9	53	284	8.56	60.5	5241	5281	8192	8047	5987	9217	291	518	60.0	46.1	22804	32748	22804								
10	3	284	7.85	60.2	5223	5202	8103	8056	5952	9222	291	517	59.8	46.1	22703	32535	22703								
10	13	284	9.01	60.4	5223	5246	8086	8038	6023	9878	292	517	59.5	46.1	22804	32615	22804								
10	23	283	8.55	60.7	5303	5281	8086	8109	5978	9615	291	520	60.0	46.0	22911	32757	22911								
10	33	284	8.63	60.6	5294	5317	8130	8091	6049	9215	292	523	60.3	46.1	22900	32881	22900								
10	43	284	8.06	60.7	5267	5272	8139	8100	6058	8619	292	522	59.9	45.9	22877	32836	22877								
10	53	284	8.86	60.9	5276	5290	8147	8126	5996	8714	293	523	60.1	45.9	22979	32836	22979								
11	3	284	8.45	61.2	5321	5325	8174	8109	6067	8711	293	524	59.5	45.9	23046	32996	23046								
11	13	285	8.79	61.4	5347	5405	8174	8179	6058	8506	293	527	59.8	46.0	23176	33164	23176								
11	23	286	9.13	61.5	5383	5370	8200	8188	6129	8584	294	529	59.9	46.0	23199	33271	23199								
11	33	286	8.87	61.6	5374	5396	8200	8188	6103	8218	294	531	59.9	46.1	23221	33262	23221								
11	43	286	8.74	61.6	5383	5388	8218	8171	6094	8115	294	532	60.2	46.0	23199	33253	23199								
11	53	287	7.86	61.6	5374	5405	8218	8188	6120	8048	295	531	60.1	45.9	23148	33306	23148								
12	3	286	8.95	61.3	5330	5388	8236	8197	6076	7938	295	528	59.6	46.0	23176	33226	23176								
Average		284.6	8.66	61.17	5324	5348	8174	8143	6060	8778	292.3	525.4	59.99	46.00	23063	33048	23063								

Hg Test CEM Data

Date:	10/20/99					
	CO2	Stack Flow	NOx	SO2	Stack T	Opacity
	%wet	wscfh68	wet ppm	wet ppm	deg F	Stack %
13:24	15.9	169202	24.0	34.0	297	0.7
13:25	15.9	170120	24.0	32.0	297	0.7
13:26	15.9	169661	26.0	32.0	297	0.8
13:27	15.9	168626	27.0	31.0	297	0.7
13:28	15.9	168395	25.0	31.0	297	0.7
13:29	15.9	171260	25.0	31.0	297	0.7
13:30	15.8	170577	25.0	31.0	297	0.7
13:31	15.8	169833	25.0	30.0	297	0.7
13:32	15.8	168510	24.0	30.0	297	0.7
13:33	15.8	167293	23.0	29.0	297	0.7
13:34	15.8	168163	22.0	29.0	297	0.7
13:35	15.9	168221	22.0	29.0	297	0.7
13:36	15.9	169661	21.0	28.0	297	0.7
13:37	15.9	170691	21.0	28.0	297	0.7
13:38	15.9	169547	20.0	29.0	297	0.7
13:39	15.9	168395	20.0	29.0	297	0.7
13:40	15.9	169432	22.0	29.0	297	0.7
13:41	15.9	167642	22.0	28.0	297	0.7
13:42	15.9	167700	22.0	28.0	297	0.7
13:43	15.9	169317	23.0	29.0	297	0.7
13:44	15.9	167468	24.0	29.0	297	0.7
13:45	15.9	169087	24.0	29.0	297	0.7
13:46	16.0	167177	23.0	29.0	297	0.7
13:47	16.0	165950	23.0	29.0	297	0.7
13:48	16.0	168221	23.0	29.0	297	0.7
13:49	16.0	168914	23.0	28.0	297	0.7
13:50	15.7	170975	24.0	28.0	297	0.7
13:51	15.8	170177	24.0	29.0	297	0.7
13:52	15.8	169719	24.0	29.0	297	0.7
13:53	15.8	167002	24.0	29.0	297	0.7
13:54	15.8	167816	22.0	29.0	297	0.7
13:55	16.0	166944	22.0	29.0	297	0.7
13:56	16.0	166886	21.0	29.0	297	0.7
13:57	16.0	166594	21.0	30.0	297	0.7
13:58	15.9	168914	23.0	30.0	297	0.7
13:59	16.0	170634	23.0	31.0	297	0.7
14:00	15.8	169374	23.0	31.0	297	0.7
14:01	15.9	169893	23.0	31.0	298	0.7
14:02	15.9	170120	24.0	31.0	297	0.7
14:03	15.9	168856	25.0	31.0	297	0.7
14:04	15.9	168510	24.0	31.0	297	0.7
14:05	15.9	167937	24.0	30.0	298	0.7
14:06	15.8	167008	23.0	30.0	298	0.7
14:07	15.9	168399	23.0	30.0	298	0.7
14:08	15.8	168918	22.0	30.0	298	0.7
14:09	15.8	171770	22.0	29.0	298	0.7
14:10	15.9	170577	22.0	29.0	297	0.7
14:11	15.8	169779	22.0	30.0	298	0.7

Hg Test CEM Data

14:12	15.8	169205	22.0	30.0	298	0.7
14:13	15.9	168918	22.0	31.0	298	0.7
14:14	15.9	167879	23.0	30.0	298	0.7
14:15	15.9	167589	24.0	30.0	298	0.7
14:16	15.9	167995	24.0	30.0	298	0.7
14:17	15.9	168687	24.0	30.0	298	0.7
14:18	15.9	170692	24.0	30.0	298	0.7
14:19	15.8	170749	23.0	30.0	298	0.7
14:20	15.8	171544	25.0	30.0	298	0.7
14:21	15.7	171374	25.0	30.0	298	0.7
14:22	15.8	170179	24.0	30.0	298	0.7
14:23	15.9	168745	23.0	31.0	298	0.7
14:24	15.9	166717	21.0	32.0	298	0.7
14:25	15.8	169779	21.0	30.0	298	0.7
14:26	15.8	171940	21.0	30.0	298	0.7
14:27	15.8	169721	21.0	29.0	298	0.7
14:28	15.8	169263	23.0	29.0	298	0.7
14:29	15.8	168399	24.0	29.0	298	0.7
14:30	15.8	169377	23.0	29.0	298	0.7
14:31	15.8	167710	23.0	29.0	299	0.7
14:32	15.8	167942	23.0	29.0	299	0.7
14:33	15.9	167421	23.0	29.0	299	0.7
14:34	15.9	167826	21.0	29.0	299	0.7
14:35	15.8	167768	21.0	29.0	299	0.7
14:36	15.9	168921	21.0	30.0	299	0.7
14:37	16.0	167595	21.0	29.0	299	0.7
14:38	15.9	167131	21.0	29.0	299	0.7
14:39	16.0	167826	22.0	29.0	299	0.7
14:40	15.9	165848	23.0	29.0	299	0.7
14:41	15.9	165438	23.0	29.0	299	0.7
14:42	15.9	165555	24.0	30.0	299	0.7
14:43	16.0	165203	24.0	29.0	299	0.7
14:44	15.9	166724	23.0	29.0	299	0.7
14:45	16.0	164379	23.0	29.0	299	0.7
14:46	16.0	165555	22.0	29.0	299	0.7
14:47	16.0	166549	22.0	31.0	299	0.7
14:48	15.9	167473	23.0	31.0	298	0.7
14:49	15.9	168457	24.0	30.0	298	0.7
14:50	15.9	167357	25.0	30.0	298	0.7
14:51	15.8	168975	25.0	30.0	298	0.7
14:52	15.8	168630	26.0	30.0	298	0.7
14:53	15.8	168918	27.0	30.0	298	0.7
14:54	15.8	168687	26.0	29.0	298	0.7
14:55	15.8	167937	25.0	29.0	298	0.7
14:56	15.8	167995	24.0	29.0	298	0.7
14:57	15.9	166542	23.0	29.0	298	0.7
14:58	16.0	167357	21.0	30.0	298	0.7
14:59	16.0	168284	21.0	30.0	298	0.7
15:00	15.8	170579	20.0	31.0	299	0.7
15:01	15.8	169781	20.0	29.0	299	0.7
15:02	15.8	168576	23.0	29.0	299	0.7

Hg Test CEM Data

15:03	15.7	168057	24.0	29.0	299	0.7
15:04	15.7	167653	23.0	29.0	299	0.7
15:05	15.8	167247	22.0	28.0	299	0.7
15:06	15.8	166724	23.0	27.0	299	0.6
15:07	15.9	166024	23.0	28.0	299	0.7
15:08	15.9	166316	23.0	29.0	299	0.7
15:09	15.9	167884	23.0	29.0	299	0.7
15:10	15.8	168634	24.0	28.0	299	0.7
15:11	15.9	168173	25.0	28.0	299	0.7
15:12	15.7	169380	25.0	27.0	299	0.6
15:13	15.8	168115	25.0	27.0	299	0.7
15:14	15.8	165965	23.0	27.0	299	0.7
15:15	15.8	165790	23.0	27.0	299	0.7
15:16	15.8	167537	22.0	28.0	299	0.7
15:17	15.9	169036	22.0	27.0	299	0.7
15:18	15.8	169895	21.0	27.0	299	0.7
15:19	15.8	169609	21.0	27.0	299	0.7
15:20	15.8	169838	23.0	27.0	299	0.7
15:21	15.8	168115	23.0	27.0	299	0.7
15:22	15.9	169380	23.0	27.0	299	0.7
15:23	15.8	167363	23.0	27.0	299	0.6
15:24	15.9	167305	22.0	27.0	299	0.6
15:25	15.9	166665	22.0	26.0	299	0.7
15:26	15.9	165144	22.0	25.0	299	0.7
15:27	15.9	166024	22.0	25.0	299	0.7
15:28	15.8	166782	23.0	26.0	299	0.6
15:29	15.8	165907	24.0	28.0	299	0.7
15:30	15.8	168115	25.0	28.0	299	0.7
15:31	15.8	169380	25.0	26.0	299	0.7
15:32	15.8	168461	24.0	25.0	299	0.7
15:33	15.8	167131	24.0	25.0	299	0.7
15:34	15.8	168346	23.0	25.0	299	0.7
15:35	15.9	167247	23.0	25.0	299	0.7
15:36	15.9	166491	23.0	25.0	299	0.7
15:37	15.9	167768	23.0	25.0	299	0.7
15:38	15.7	170466	21.0	25.0	299	0.7
15:39	15.8	169208	20.0	25.0	299	0.7
15:40	15.8	171544	22.0	24.0	299	0.7
15:41	15.8	170238	23.0	25.0	299	0.7
15:42	15.8	168523	23.0	25.0	300	0.7
15:43	15.8	167658	24.0	24.0	300	0.7
15:44	15.7	166730	23.0	24.0	300	0.7
15:45	15.8	167600	23.0	24.0	300	0.7
15:46	15.8	167253	22.0	24.0	300	0.6
15:47	15.8	168235	22.0	24.0	300	0.6
15:48	15.8	168925	24.0	24.0	300	0.7
15:49	15.7	170467	24.0	23.0	300	0.6
15:50	15.7	169669	25.0	23.0	300	0.7
15:51	15.8	168982	25.0	23.0	300	0.7
15:52	15.8	169498	24.0	23.0	300	0.7
15:53	15.8	166323	24.0	23.0	300	0.6

Hg Test CEM Data

15:54	15.8	166614	23.0	24.0	300	0.7
15:55	15.9	165798	23.0	24.0	300	0.7
15:56	15.9	169097	22.0	23.0	300	0.6
15:57	15.8	171148	22.0	24.0	300	0.7
15:58	15.7	169669	23.0	25.0	300	0.7
15:59	15.7	169841	23.0	24.0	300	0.7
16:00	15.7	170069	22.0	23.0	300	0.7
16:01	15.6	167947	22.0	23.0	300	0.7
16:02	15.7	166905	21.0	23.0	300	0.7
16:03	15.8	168235	21.0	24.0	300	0.7
16:04	15.9	169097	20.0	24.0	300	0.7
16:05	15.9	172839	20.0	23.0	300	0.7
16:06	15.9	170297	21.0	23.0	300	0.7
16:07	15.9	170865	21.0	24.0	300	0.7
16:08	15.9	168293	20.0	24.0	300	0.7
16:09	15.9	168580	20.0	22.0	300	0.7
16:10	16.0	168120	22.0	22.0	300	0.7
16:11	15.9	167079	22.0	23.0	300	0.7
16:12	15.8	170069	21.0	24.0	300	0.7
16:13	15.9	168408	21.0	22.0	300	0.7
16:14	15.9	167542	22.0	22.0	300	0.7
16:15	15.9	167836	22.0	22.0	301	0.7
16:16	15.8	168355	21.0	22.0	301	0.7
16:17	15.9	169329	21.0	22.0	301	0.7
16:18	15.8	167836	20.0	21.0	301	0.7
16:19	15.9	167663	20.0	21.0	301	0.6
16:20	15.8	168182	20.0	21.0	301	0.7
16:21	15.9	167952	19.0	22.0	301	0.7
16:22	15.9	165572	21.0	23.0	301	0.7
16:23	16.0	163513	21.0	22.0	301	0.7
16:24	15.9	163572	18.0	22.0	301	0.7
16:25	16.0	161787	17.0	21.0	301	0.7
16:26	16.1	161187	18.0	20.0	301	0.6
16:27	16.1	161547	19.0	22.0	301	0.7
16:28	16.1	161846	18.0	22.0	301	0.7
16:29	16.1	161727	18.0	22.0	301	0.7
16:30	16.1	162205	19.0	22.0	301	0.7
16:31	16.2	160525	20.0	22.0	301	0.6
16:32	16.1	160887	21.0	22.0	301	0.7
16:33	16.1	162503	22.0	21.0	301	0.7
16:34	16.0	164280	23.0	20.0	301	0.7
16:35	16.0	163739	23.0	21.0	300	0.7
16:36	16.0	163205	23.0	22.0	300	0.7
16:37	15.9	162371	24.0	21.0	300	0.7
16:38	16.0	164093	23.0	21.0	300	0.7
16:39	16.0	163146	23.0	22.0	300	0.7
16:40	16.0	163739	22.0	22.0	300	0.7
Hg1 Avg	15.87	167865	22.56	26.97	298.8	0.69

Hg Test CEM Data

Date:	10/21/99					
	CO2	Stack Flow	NOx	SO2	Stack T	Opacity
	%wet	wscfh68	wet ppm	wet ppm	deg F	Stack %
Date:	21-Oct					
10:12	16.2	164707	27.0	21.0	290	0.8
10:13	16.2	164469	27.0	20.0	290	0.8
10:14	16.2	166664	28.0	20.0	290	0.8
10:15	16.1	164767	28.0	20.0	290	0.8
10:16	16.2	165599	30.0	20.0	290	0.8
10:17	16.2	166014	30.0	22.0	290	0.8
10:18	16.1	168189	31.0	22.0	290	0.8
10:19	16.3	166840	31.0	21.0	290	0.8
10:20	16.2	165184	31.0	22.0	290	0.8
10:21	16.2	164409	31.0	22.0	290	0.8
10:22	16.3	164230	31.0	22.0	290	0.8
10:23	16.2	164478	31.0	23.0	291	0.8
10:24	16.3	164955	29.0	23.0	291	0.8
10:25	16.3	164776	28.0	24.0	291	0.8
10:26	16.3	165074	28.0	25.0	291	0.8
10:27	16.3	168368	28.0	26.0	291	0.8
10:28	16.3	168019	30.0	26.0	291	0.8
10:29	16.1	166729	32.0	26.0	291	0.8
10:30	16.1	167082	33.0	26.0	291	0.8
10:31	16.1	167316	34.0	26.0	291	0.8
10:32	16.1	166494	33.0	27.0	291	0.8
10:33	16.1	166258	33.0	28.0	291	0.8
10:34	16.1	165608	32.0	28.0	291	0.8
10:35	16.1	166670	31.0	27.0	291	0.8
10:36	16.1	166847	30.0	27.0	291	0.8
10:37	16.1	166376	29.0	29.0	291	0.8
10:38	16.1	165726	27.0	29.0	291	0.8
10:39	16.0	165014	27.0	28.0	291	0.8
10:40	16.1	165371	27.0	27.0	291	0.8
10:41	16.1	166206	27.0	29.0	292	0.8
10:42	16.1	163712	26.0	29.0	292	0.8
10:43	15.9	166736	27.0	29.0	292	0.8
10:44	16.0	166088	28.0	29.0	292	0.8
10:45	15.9	165092	28.0	28.0	293	0.8
10:46	16.0	164141	29.0	28.0	293	0.8
10:47	16.0	162102	30.0	26.0	293	0.8
10:48	16.1	163076	30.0	26.0	294	0.8
10:49	16.1	162175	30.0	26.0	294	0.8
10:50	16.2	161390	30.0	27.0	294	0.8
10:51	16.2	163088	30.0	26.0	295	0.8
10:52	16.1	162668	30.0	26.0	295	0.8
10:53	16.1	160510	30.0	28.0	296	0.8
10:54	16.0	160570	27.0	29.0	296	0.8
10:55	16.1	160646	27.0	28.0	297	0.8
10:56	16.2	160464	27.0	28.0	297	0.8
10:57	16.3	157712	27.0	29.0	297	0.8
10:58	16.3	157423	30.0	30.0	298	0.8

Hg Test CEM Data

10:59	16.3	157114	31.0	27.0	298	0.8
11:00	16.4	159386	32.0	25.0	298	0.8
11:01	16.3	159690	32.0	28.0	298	0.8
11:02	16.3	159751	31.0	29.0	298	0.8
11:03	16.3	159994	31.0	28.0	298	0.8
11:04	16.4	159994	28.0	28.0	298	0.8
11:05	16.4	161686	27.0	30.0	298	0.8
11:06	16.5	160601	28.0	31.0	298	0.8
11:07	16.4	163539	28.0	30.0	298	0.8
11:08	16.4	162932	32.0	30.0	297	0.8
11:09	16.3	162693	33.0	30.0	297	0.8
11:10	16.3	162573	35.0	30.0	297	0.8
11:11	16.3	162992	36.0	29.0	297	0.8
11:12	16.3	162992	31.0	29.0	297	0.8
11:13	16.3	164290	29.0	30.0	296	0.8
11:14	16.3	164764	29.0	30.0	296	0.8
11:15	16.3	162141	29.0	30.0	296	0.8
11:16	16.3	161585	29.0	30.0	295	0.8
11:17	16.3	163207	28.0	29.0	295	0.8
11:18	16.3	163625	27.0	29.0	295	0.8
11:19	16.3	165581	27.0	31.0	295	0.8
11:20	16.3	165750	31.0	31.0	294	0.8
11:21	16.2	164448	32.0	31.0	294	0.8
11:22	16.1	164626	34.0	31.0	294	0.8
11:23	16.2	164032	34.0	31.0	294	0.8
11:24	16.2	164091	32.0	31.0	294	0.8
11:25	16.2	164151	32.0	30.0	294	0.8
11:26	16.1	164448	32.0	30.0	294	0.8
11:27	15.9	166045	31.0	30.0	294	0.7
11:28	16.1	167451	30.0	30.0	294	0.8
11:29	16.1	163734	29.0	29.0	294	0.7
11:30	16.1	163016	28.0	28.0	294	0.8
11:31	16.2	163555	27.0	29.0	294	0.8
11:32	16.2	164567	27.0	29.0	294	0.7
11:33	16.2	166690	26.0	29.0	294	0.8
11:34	16.1	165041	30.0	30.0	294	0.7
11:35	16.1	163495	31.0	30.0	294	0.8
11:36	16.1	163004	32.0	29.0	293	0.7
11:37	16.1	164021	32.0	29.0	293	0.8
11:38	16.1	167153	32.0	30.0	293	0.8
11:39	16.0	166508	32.0	29.0	293	0.8
11:40	16.0	166155	35.0	29.0	293	0.8
11:41	16.0	165269	36.0	29.0	293	0.7
11:42	16.0	163723	33.0	28.0	293	0.7
11:43	16.0	165092	32.0	29.0	293	0.7
11:44	16.0	166508	30.0	30.0	293	0.7
11:45	15.9	165742	30.0	29.0	293	0.7
11:46	16.0	163124	28.0	29.0	293	0.8
11:47	16.0	162776	27.0	27.0	294	0.7
11:48	16.1	162716	27.0	27.0	294	0.7
11:49	16.0	165160	27.0	27.0	294	0.8

Hg Test CEM Data

11:50	15.9	166983	30.0	27.0	294	0.7
11:51	15.9	167217	31.0	26.0	294	0.7
11:52	15.8	165041	32.0	26.0	294	0.7
11:53	15.9	166162	32.0	27.0	294	0.8
11:54	16.0	166045	30.0	27.0	294	0.7
11:55	16.0	165041	30.0	27.0	294	0.7
11:56	16.0	164032	29.0	27.0	294	0.7
11:57	16.0	166573	28.0	27.0	294	0.7
11:58	16.0	165927	28.0	27.0	294	0.7
11:59	16.0	166925	28.0	27.0	294	0.7
12:00	16.0	163853	30.0	27.0	294	0.7
12:01	15.9	163495	31.0	27.0	294	0.7
12:02	16.0	162054	32.0	26.0	294	0.7
12:03	16.0	165160	31.0	27.0	294	0.7
12:04	15.9	166866	31.0	27.0	294	0.7
12:05	15.9	167392	31.0	28.0	294	0.7
12:06	15.9	165160	31.0	29.0	294	0.7
12:07	15.9	164626	31.0	28.0	294	0.7
12:08	15.9	166221	31.0	28.0	294	0.7
12:09	15.9	169884	31.0	26.0	294	0.7
12:10	15.8	167917	32.0	26.0	294	0.7
12:11	15.8	167626	33.0	26.0	294	0.7
12:12	15.9	168498	30.0	27.0	294	0.7
12:13	15.9	165278	30.0	27.0	294	0.7
12:14	15.9	164508	28.0	27.0	294	0.7
12:15	15.8	166639	27.0	26.0	295	0.7
12:16	15.9	168386	27.0	26.0	295	0.7
12:17	15.8	166814	27.0	27.0	295	0.7
12:18	15.8	166580	29.0	27.0	295	0.7
12:19	15.9	166873	30.0	28.0	295	0.7
12:20	15.9	168038	31.0	28.0	295	0.7
12:21	15.8	166639	32.0	27.0	295	0.7
12:22	15.7	168096	31.0	27.0	295	0.7
12:23	15.6	167223	31.0	25.0	295	0.7
12:24	15.6	166756	31.0	25.0	295	0.7
12:25	15.7	166873	31.0	27.0	295	0.7
12:26	15.7	166170	31.0	28.0	295	0.7
12:27	15.7	167223	31.0	28.0	295	0.7
12:28	15.8	167106	29.0	28.0	295	0.7
12:29	15.7	166521	28.0	29.0	295	0.7
12:30	15.7	166404	27.0	29.0	295	0.7
12:31	15.8	165590	26.0	28.0	296	0.7
12:32	15.8	163755	26.0	28.0	296	0.7
12:33	15.8	163398	26.0	28.0	296	0.7
12:34	16.0	162621	26.0	28.0	296	0.7
12:35	15.9	162681	26.0	27.0	296	0.7
12:36	15.9	161960	28.0	27.0	296	0.7
12:37	15.9	162621	29.0	28.0	296	0.7
12:38	15.9	162381	30.0	29.0	296	0.7
12:39	15.9	163159	30.0	27.0	296	0.7
12:40	15.9	164349	33.0	27.0	296	0.7

Hg Test CEM Data

12:41	15.8	163219	34.0	28.0	296	0.7
12:42	15.9	161237	32.0	28.0	296	0.7
12:43	16.0	163755	32.0	28.0	296	0.7
12:44	15.9	163517	30.0	27.0	296	0.7
12:45	15.9	163755	29.0	26.0	296	0.7
12:46	16.0	162980	29.0	26.0	296	0.7
12:47	16.0	162681	29.0	27.0	296	0.7
12:48	16.0	163159	27.0	27.0	296	0.7
12:49	16.0	164290	27.0	28.0	296	0.7
12:50	15.9	165766	28.0	29.0	296	0.7
12:51	15.9	165354	28.0	27.0	296	0.7
12:52	16.0	165766	29.0	27.0	296	0.7
12:53	16.0	165236	30.0	27.0	296	0.7
12:54	16.0	163278	30.0	27.0	296	0.7
12:55	15.9	164941	30.0	28.0	296	0.7
12:56	16.0	164882	30.0	29.0	296	0.7
12:57	16.0	163983	31.0	29.0	295	0.7
12:58	16.0	164517	31.0	29.0	295	0.7
12:59	16.0	162668	30.0	28.0	295	0.7
13:00	16.0	164695	31.0	28.0	295	0.7
13:01	16.1	162668	31.0	29.0	295	0.7
13:02	16.0	162248	33.0	29.0	295	0.7
13:03	15.9	162788	34.0	29.0	295	0.7
13:04	16.0	164458	32.0	28.0	295	0.7
13:05	16.0	164458	32.0	29.0	295	0.7
13:06	15.9	164695	31.0	29.0	295	0.7
13:07	15.9	162488	31.0	29.0	295	0.7
13:08	16.0	163506	28.0	28.0	295	0.7
13:09	16.0	165758	28.0	29.0	295	0.7
13:10	16.0	166404	27.0	29.0	295	0.7
13:11	15.9	163864	26.0	29.0	295	0.7
13:12	15.9	162368	26.0	29.0	295	0.7
13:13	16.0	164220	27.0	29.0	295	0.7
13:14	16.0	163147	26.0	29.0	295	0.7
13:15	16.0	163387	26.0	27.0	295	0.7
13:16	16.0	161766	29.0	26.0	295	0.7
13:17	15.9	161404	29.0	28.0	295	0.7
13:18	16.1	162848	33.0	29.0	295	0.7
13:19	16.0	164823	35.0	29.0	296	0.7
13:20	15.9	166937	38.0	29.0	296	0.7
13:21	15.9	164704	39.0	29.0	296	0.7
13:22	16.0	164704	36.0	29.0	296	0.7
13:23	16.0	162860	35.0	29.0	296	0.7
13:24	16.0	163577	29.0	29.0	296	0.7
13:25	16.0	162920	27.0	29.0	296	0.7
Hg2 Avg	16.0	164460	29.95	27.61	294.3	0.74

Hg Test CEM Data

Date:	10/22/99					
	CO2	Stack Flow	NOx	SO2	Stack T	Opacity
	%wet	wscfh68	wet ppm	wet ppm	deg F	Stack %
8:50	15.8	175337	19.0	33.0	290	0.9
8:51	15.8	173931	19.0	32.0	290	0.9
8:52	15.9	173308	19.0	32.0	290	0.9
8:53	15.9	175561	20.0	33.0	290	0.9
8:54	15.8	177287	20.0	33.0	290	0.9
8:55	15.9	177508	21.0	32.0	290	0.9
8:56	15.8	175673	21.0	32.0	290	0.9
8:57	15.7	175505	22.0	32.0	290	0.9
8:58	15.8	175896	21.0	32.0	290	0.9
8:59	15.8	175896	20.0	32.0	290	0.9
9:00	15.8	176454	20.0	33.0	290	0.9
9:01	15.8	178666	21.0	32.0	290	0.9
9:02	15.7	177397	20.0	33.0	290	0.9
9:03	15.7	178721	20.0	35.0	290	0.9
9:04	15.8	177009	20.0	35.0	290	0.9
9:05	15.8	177287	19.0	32.0	290	0.9
9:06	15.8	176676	19.0	31.0	290	0.9
9:07	15.9	173874	19.0	32.0	290	0.9
9:08	15.8	175169	20.0	32.0	290	0.9
9:09	15.8	175784	20.0	32.0	290	0.9
9:10	15.8	178776	20.0	32.0	290	0.9
9:11	15.7	178776	20.0	32.0	290	0.9
9:12	15.7	177508	20.0	32.0	290	0.9
9:13	15.7	178005	20.0	33.0	290	0.9
9:14	15.7	178666	21.0	33.0	290	0.9
9:15	15.7	178556	21.0	33.0	290	0.9
9:16	15.8	176565	20.0	33.0	290	0.9
9:17	15.8	176175	20.0	33.0	290	0.9
9:18	15.9	175835	20.0	32.0	291	0.9
9:19	15.9	174603	19.0	33.0	291	0.9
9:20	15.9	173419	19.0	34.0	291	0.9
9:21	15.9	174772	19.0	34.0	291	0.9
9:22	15.8	176392	19.0	34.0	291	0.8
9:23	15.8	177942	19.0	33.0	291	0.9
9:24	15.8	177002	20.0	33.0	291	0.9
9:25	15.9	173702	21.0	33.0	291	0.9
9:26	15.9	175388	20.0	33.0	291	0.9
9:27	15.8	176781	20.0	32.0	291	0.9
9:28	15.9	175052	21.0	33.0	291	0.9
9:29	15.8	173533	21.0	33.0	291	0.9
9:30	15.8	172683	20.0	34.0	291	0.9
9:31	15.8	173871	20.0	34.0	291	0.9
9:32	15.8	173306	20.0	34.0	291	0.9
9:33	15.8	173871	20.0	33.0	291	0.9
9:34	15.9	171828	19.0	33.0	291	0.8
9:35	15.8	171027	19.0	34.0	291	0.9
9:36	15.8	170798	19.0	35.0	291	0.9
9:37	15.9	171885	19.0	33.0	291	0.9

Hg Test CEM Data

9:38	15.8	169182	19.0	32.0	291	0.9
9:39	15.9	169240	18.0	34.0	291	0.9
9:40	15.9	168659	18.0	34.0	291	0.8
9:41	15.9	167199	18.0	33.0	291	0.9
9:42	15.9	168427	18.0	34.0	291	0.9
9:43	16.0	169356	18.0	33.0	291	0.9
9:44	15.9	170223	19.0	33.0	291	0.9
9:45	15.9	170798	19.0	34.0	291	0.9
9:46	15.9	168485	20.0	34.0	291	0.9
9:47	15.9	169876	20.0	33.0	291	0.9
9:48	15.9	171657	21.0	33.0	291	0.9
9:49	15.9	172228	21.0	33.0	291	0.9
9:50	15.9	169992	20.0	33.0	291	0.9
9:51	15.9	168135	20.0	32.0	291	0.9
9:52	16.0	169298	20.0	31.0	291	0.8
9:53	16.0	168834	20.0	31.0	291	0.9
9:54	15.9	168547	19.0	31.0	292	0.9
9:55	15.9	170971	19.0	34.0	292	0.9
9:56	15.9	169414	21.0	35.0	291	0.9
9:57	15.9	169934	22.0	33.0	291	0.9
9:58	16.0	169761	24.0	32.0	291	0.9
9:59	15.9	168834	25.0	32.0	291	0.9
10:00	15.9	167902	27.0	32.0	291	0.9
10:01	15.9	166317	28.0	32.0	291	0.9
10:02	15.9	166081	30.0	32.0	291	0.9
10:03	16.0	166081	31.0	31.0	291	0.9
10:04	16.0	168659	31.0	31.0	291	0.9
10:05	16.0	168427	31.0	31.0	291	0.9
10:06	16.0	168135	31.0	31.0	291	0.8
10:07	16.0	168431	32.0	31.0	292	0.9
10:08	16.0	167147	30.0	32.0	292	0.9
10:09	15.9	169475	29.0	31.0	292	0.9
10:10	15.9	168315	29.0	31.0	292	0.8
10:11	16.0	167322	28.0	31.0	292	0.8
10:12	16.0	168023	27.0	31.0	292	0.8
10:13	15.9	168896	26.0	31.0	292	0.9
10:14	16.0	169475	26.0	31.0	292	0.9
10:15	15.8	169070	26.0	31.0	292	0.9
10:16	15.9	169301	27.0	31.0	292	0.9
10:17	15.9	167907	27.0	32.0	292	0.8
10:18	15.9	168718	27.0	32.0	291	0.9
10:19	16.0	167785	26.0	31.0	291	0.8
10:20	15.9	169819	28.0	30.0	291	0.8
10:21	15.9	170855	28.0	30.0	291	0.8
10:22	15.9	168892	31.0	30.0	291	0.8
10:23	15.9	167668	31.0	30.0	291	0.8
10:24	15.9	169992	32.0	30.0	291	0.8
10:25	15.9	171257	32.0	29.0	291	0.8
10:26	15.9	171714	31.0	28.0	291	0.8
10:27	15.8	170855	30.0	30.0	291	0.8
10:28	15.8	170107	29.0	31.0	291	0.8

Hg Test CEM Data

10:29	15.8	170397	28.0	31.0	292	0.8
10:30	15.8	171143	28.0	31.0	292	0.8
10:31	15.9	170109	28.0	29.0	292	0.8
10:32	15.9	166912	27.0	28.0	292	0.8
10:33	16.0	167965	26.0	29.0	292	0.8
10:34	15.8	168547	26.0	29.0	292	0.8
10:35	15.8	169070	26.0	29.0	292	0.8
10:36	15.8	169417	26.0	29.0	292	0.8
10:37	15.8	169243	27.0	28.0	292	0.8
10:38	15.8	169012	27.0	27.0	292	0.8
10:39	15.8	167498	28.0	30.0	292	0.8
10:40	15.8	166618	26.0	30.0	292	0.8
10:41	15.9	166618	25.0	30.0	292	0.8
10:42	15.9	168373	27.0	30.0	292	0.8
10:43	15.9	168023	28.0	30.0	292	0.8
10:44	15.9	166147	29.0	29.0	292	0.8
10:45	15.9	165793	29.0	29.0	292	0.8
10:46	15.9	166331	28.0	29.0	293	0.8
10:47	15.9	167153	29.0	29.0	293	0.8
10:48	15.7	169881	30.0	30.0	293	0.8
10:49	15.8	168668	30.0	29.0	293	0.8
10:50	15.8	167620	29.0	29.0	293	0.8
10:51	15.8	168668	29.0	28.0	293	0.8
10:52	15.8	167211	29.0	28.0	293	0.8
10:53	15.8	168493	29.0	28.0	293	0.8
10:54	15.8	167503	27.0	28.0	293	0.8
10:55	15.9	168145	27.0	29.0	293	0.8
10:56	15.9	168783	27.0	29.0	293	0.8
10:57	15.9	168551	27.0	28.0	293	0.8
10:58	15.9	168028	27.0	28.0	293	0.8
10:59	15.8	167445	27.0	29.0	293	0.8
11:00	15.9	167270	26.0	30.0	293	0.8
11:01	15.9	167970	26.0	29.0	293	0.8
11:02	15.8	169593	29.0	29.0	293	0.8
11:03	15.8	169881	30.0	29.0	293	0.8
11:04	15.8	169304	30.0	29.0	293	0.8
11:05	15.7	169304	30.0	30.0	293	0.7
11:06	15.7	170456	29.0	30.0	293	0.8
11:07	15.7	171657	29.0	29.0	293	0.8
11:08	15.7	170226	28.0	29.0	293	0.8
11:09	15.7	168725	28.0	29.0	293	0.8
11:10	15.9	169420	27.0	29.0	293	0.8
11:11	15.9	168145	27.0	28.0	293	0.8
11:12	15.9	169651	26.0	28.0	293	0.8
11:13	15.7	169826	26.0	27.0	294	0.8
11:14	15.8	173581	28.0	28.0	294	0.7
11:15	15.7	172622	29.0	29.0	294	0.7
11:16	15.7	170572	29.0	29.0	294	0.7
11:17	15.7	170687	29.0	28.0	294	0.8
11:18	15.7	170171	30.0	28.0	294	0.7
11:19	15.8	171372	30.0	29.0	294	0.7

Hg Test CEM Data

11:20	15.8	169596	28.0	29.0	294	0.8
11:21	15.8	170916	27.0	30.0	294	0.8
11:22	15.9	170916	26.0	30.0	294	0.7
11:23	15.8	171315	26.0	27.0	294	0.8
11:24	15.8	171998	27.0	26.0	294	0.7
11:25	15.7	171771	27.0	28.0	294	0.7
11:26	15.7	173187	28.0	28.0	294	0.7
11:27	15.7	173412	28.0	28.0	294	0.7
11:28	15.7	174648	29.0	27.0	294	0.7
11:29	15.7	171543	29.0	27.0	294	0.7
11:30	15.8	172112	29.0	28.0	294	0.7
11:31	15.7	171315	29.0	29.0	294	0.7
11:32	15.7	170744	28.0	29.0	294	0.7
11:33	15.8	172905	27.0	30.0	294	0.7
11:34	15.8	173694	28.0	31.0	294	0.7
11:35	15.7	175318	29.0	29.0	294	0.8
11:36	15.7	175094	28.0	29.0	294	0.7
11:37	15.6	172792	28.0	29.0	294	0.7
11:38	15.7	172169	27.0	29.0	294	0.7
11:39	15.7	169941	25.0	28.0	294	0.8
11:40	15.8	171258	27.0	28.0	294	0.8
11:41	15.8	172792	27.0	28.0	294	0.8
11:42	15.7	173356	28.0	28.0	294	0.8
11:43	15.7	173356	29.0	30.0	294	0.8
11:44	15.7	173131	28.0	31.0	294	0.8
11:45	15.7	174087	27.0	30.0	294	0.8
11:46	15.6	174592	29.0	30.0	294	0.7
11:47	15.6	174983	29.0	30.0	294	0.8
11:48	15.7	173298	30.0	29.0	295	0.7
11:49	15.6	175368	30.0	29.0	295	0.7
11:50	15.5	174028	29.0	30.0	295	0.8
11:51	15.5	173916	28.0	29.0	295	0.7
11:52	15.6	172111	27.0	29.0	295	0.7
11:53	15.7	172903	27.0	30.0	295	0.7
11:54	15.7	172508	27.0	30.0	295	0.7
11:55	15.7	172281	27.0	28.0	295	0.7
11:56	15.7	172847	26.0	28.0	295	0.7
11:57	15.7	170802	26.0	30.0	295	0.8
11:58	15.8	170402	27.0	30.0	295	0.8
11:59	15.7	171600	27.0	29.0	295	0.8
12:00	15.7	171088	27.0	29.0	295	0.8
Hg3 Avg	15.8	171420	25.1	30.7	292.1	0.8

Appendix C.3

Gaseous Emissions (O₂ and CO₂) Data

Injection of Orsat Bags

	O ₂ %	CO ₂ %	
10-22-1999 12:58:52	2.020	4.527	
Start Averaging			
10-22-1999 12:59:53	0.000	0.066	
Average 35 samples	-0.002	0.062	Pre Bias (zero)
10-22-1999 13:00:53	1.838	4.770	
Start Averaging			
10-22-1999 13:01:54	5.069	14.013	
Average 31 samples	5.068	14.020	Pre Bias (span)
10-22-1999 13:02:54	4.060	10.390	
10-22-1999 13:03:54	2.295	6.663	
Start Averaging			
10-22-1999 13:04:53	5.110	14.535	
Average 30 samples	5.109	14.536	1-Hg-O
10-22-1999 13:05:53	1.857	5.075	
10-22-1999 13:06:53	3.492	5.357	
10-22-1999 13:07:54	3.009	8.413	
Start Averaging			
10-22-1999 13:08:54	5.257	14.502	
Average 31 samples	5.256	14.502	2-Hg-O
10-22-1999 13:09:52	1.282	3.220	
Start Averaging			
10-22-1999 13:10:53	3.620	10.495	
Average 31 samples	5.190	14.493	3-Hg-O
10-22-1999 13:11:53	5.190	14.492	
10-22-1999 13:12:53	1.598	4.112	
Start Averaging			
10-22-1999 13:13:54	2.855	12.342	
10-22-1999 13:14:54	3.723	15.806	
Average 30 samples	3.723	15.806	1-Hg-I
10-22-1999 13:15:52	0.487	1.601	
Start Averaging			
10-22-1999 13:16:53	4.270	14.881	
Average 30 samples	4.395	14.984	2-Hg-I
10-22-1999 13:17:53	4.394	14.983	
10-22-1999 13:18:53	0.799	2.892	
Start Averaging			
10-22-1999 13:19:54	3.865	15.717	
Average 31 samples	3.891	15.748	3-Hg-I
10-22-1999 13:20:52	2.822	10.923	
Start Averaging			
10-22-1999 13:21:53	-0.012	0.140	
Average 34 samples	-0.017	0.115	Post Bias (zero)
10-22-1999 13:22:53	-0.017	0.113	
10-22-1999 13:23:53	4.581	12.905	
Start Averaging			
10-22-1999 13:24:54	5.044	14.015	
Average 33 samples	5.044	14.017	Post Bias (span)

CEM System Bias and Linearity Correction Calculations

Gas Species	O ₂	CO ₂	O ₂	CO ₂	O ₂	CO ₂	Criteria	Status	
Test #	1-Hg-O		2-Hg-O		3-Hg-O				
Stockton CoGen							Time: 1258-1325		
Date: 10/22/99							Unit: Outlet		
LINEARITY									
Analyzer Range:	10	25	10	25	10	25			
High Cal.	7.92	20.78	7.92	20.78	7.92	20.78			
Low Cal.	5.02	14.02	5.02	14.02	5.02	14.02			
Analyzer Reads:	5.04	14.05	5.04	14.05	5.04	14.05			
Anal. Cal. Error:	0.2	0.1	0.2	0.1	0.2	0.1	< 2.0%	PASS	
SYSTEM BIAS									
Bias Span Value:	5.02	14.02	5.02	14.02	5.02	14.02			
Pre-test Bias									
Zero:	0.00	0.06	0.00	0.06	0.00	0.06			
Span:	5.07	14.02	5.07	14.02	5.07	14.02			
Zero Bias, %:	-0.02	0.25	-0.02	0.25	-0.02	0.25	< 5.0%		PASS
Span Bias, %:	0.48	0.00	0.48	0.00	0.48	0.00	< 5.0%		PASS
Zero Drift, %:	-0.02	0.25	-0.02	0.25	-0.02	0.25	< 3.0%		PASS
Span Drift, %:	0.48	0.00	0.48	0.00	0.48	0.00	< 3.0%		PASS
Post-test Bias									
Zero:	-0.02	0.12	-0.02	0.12	-0.02	0.12			
Span:	5.04	14.02	5.04	14.02	5.04	14.02			
Zero Bias, %:	-0.17	0.46	-0.17	0.46	-0.17	0.46	< 5.0%	PASS	
Span Bias, %:	0.24	-0.01	0.24	-0.01	0.24	-0.01	< 5.0%	PASS	
Zero Drift, %:	-0.15	0.21	-0.15	0.21	-0.15	0.21	< 3.0%	PASS	
Span Drift, %:	-0.24	-0.01	-0.24	-0.01	-0.24	-0.01	< 3.0%	PASS	
Test Ave.	5.11	14.54	5.26	14.50	5.19	14.49			
Corrected Ave.	5.07	14.54	5.22	14.51	5.15	14.50			

CEM System Bias and Linearity Correction Calculations

Gas Species	O ₂	CO ₂	O ₂	CO ₂	O ₂	CO ₂	Criteria	Status	
Test #	1-Hg-I	2-Hg-I	3-Hg-I						
Stockton CoGen							Time: 1258-1325		
Date: 10/22/99							Unit: Inlet		
LINEARITY									
Analyzer Range:	10	25	10	25	10	25			
High Cal.	7.92	20.78	7.92	20.78	7.92	20.78			
Low Cal.	5.02	14.02	5.02	14.02	5.02	14.02			
Analyzer Reads:	5.04	14.05	5.04	14.05	5.04	14.05			
Anal. Cal. Error:	0.2	0.1	0.2	0.1	0.2	0.1	< 2.0%	PASS	
SYSTEM BIAS									
Bias Span Value:	5.02	14.02	5.02	14.02	5.02	14.02			
Pre-test Bias									
Zero:	0.00	0.06	0.00	0.06	0.00	0.06			
Span:	5.07	14.02	5.07	14.02	5.07	14.02			
Zero Bias, %:	-0.02	0.25	-0.02	0.25	-0.02	0.25	< 5.0%		PASS
Span Bias, %:	0.48	0.00	0.48	0.00	0.48	0.00	< 5.0%		PASS
Zero Drift, %:	-0.02	0.25	-0.02	0.25	-0.02	0.25	< 3.0%		PASS
Span Drift, %:	0.48	0.00	0.48	0.00	0.48	0.00	< 3.0%		PASS
Post-test Bias									
Zero:	-0.02	0.12	-0.02	0.12	-0.02	0.12			
Span:	5.04	14.02	5.04	14.02	5.04	14.02			
Zero Bias, %:	-0.17	0.46	-0.17	0.46	-0.17	0.46	< 5.0%		PASS
Span Bias, %:	0.24	-0.01	0.24	-0.01	0.24	-0.01	< 5.0%		PASS
Zero Drift, %:	-0.15	0.21	-0.15	0.21	-0.15	0.21	< 3.0%		PASS
Span Drift, %:	-0.24	-0.01	-0.24	-0.01	-0.24	-0.01	< 3.0%		PASS
Test Ave.	3.72	15.81	4.40	14.98	3.89	15.75			
Corrected Ave.	3.70	15.82	4.36	14.99	3.87	15.76			

Appendix C.4

Isokinetic Sampling Data

SAMPLE TRAIN TEST DATA

CLIENT Air Products & Stockton Cogen TEST NO. 1-Hg-0 METHOD Ontario Hydro PAGE 1 OF 2
 SAMPLE LOCATION Stockton/Boiler Outlet Ports UNIT Boiler AMB. TEMP., °F 75 PROJECT # 99057
 OPERATOR/ASSISTANT Peter Gates METER VOL. (START/END) 192.669 / 277.745 DATE 10-20-99

PRE-TEST DATA:

Barometric Press., in. Hg. 29.95
 Assumed Slack Press. -80
 Assumed Moisture 5.5
 Assumed Molecular Wt. 39.85
 Assumed ΔP 0.90
 Assumed ΔH 1.20
 Stack Diameter, in. 54 ft.
 Sample Time: Total 50.37
 per point 1.60
 Total of Traverse Points 16

 $\Delta H = 0.6 \times \Delta P$

EQUIPMENT INFO:

Meter No. N-3
 Meter, Yd. 0.996
 CFM @ ΔH 1.786
 Pilot ID, Cp 0-108 0.84
 O₂/CO₂ Method CEM
 Teflon Connecting Y
 Line (Y/N) Y
 Probe: Mat'l G-1555
 Length 10"
 Nozzle: Mat'l G-1555
 Diam. 1.11"
 Filler: No. N/A
 Mat'l Quartz

IMP. Mat'l WL(End) WL(Start) WL(Q)

#1 100% kcl sln 688.9 634.4 54.5
 #2 " 6633 633.2 30.1
 #3 " 623.0 619.7 3.3
 #4 100% kcl sln 623.7 621.6 2.1
 #5 100% kcl sln 635.7 636.2 -0.5

Total

POST TEST INFO:

Filter Appearance

Impinger Appearance

Silica Gel Spent (Y/N)

(continued)

Compage 2

G-1555

Clear

No

SAMPLE TRAIN LEAK CHECK:

CEM Vac. Pilot Inlet
 Pre-Test 0.014 -19 OK
 Post-Test 0.012 -17 OK

PRE-TEST CALIBRATION CHECK:

Meter Meter Temp.
 Time ΔH Reading In Out
 Init. --- --- ---
 Final --- --- ---

SAMPLE POINT	TIME	METER CONDITIONS			TEMPERATURES, °F				Heated Line	STATIC PRESS. lwg	CHAIN OF CUSTODY INFORMATION	
		ΔP	ΔH	METER READING	PROBE	METER IN	METER OUT	OVEN	IMP. OUT	VAC.	Impingers Loaded	Impingers Recovered
8	1330	1.25	0.75	192.669	290	73	72	296	45	-2	DD	DD
	1335	1.25	0.75	195.124	290	84	77	294	45	-2	EM	EM
7	1340	1.38	0.83	197.893	291	85	78	293	44	-2	DD	DD
	1345	1.38	0.83	199.886	289	92	80	294	43	-2	DD	DD
6	1350	1.55	0.93	202.455	293	94	81	293	43	-2	RG	RG
	1355	1.55	0.93	205.251	294	95	83	291	42	-2	TEST SUMMARY	TEST SUMMARY
5	1400	1.51	0.91	208.037	291	97	84	292	43	-2	Calculated by: <u>RK</u>	Calculated by: <u>RK</u>
	1405	1.51	0.91	210.629	295	99	86	293	43	-2	Checked by: <u>EM</u>	Checked by: <u>EM</u>
4	1410	1.66	1.0	213.372	295	101	88	292	42	-2	Sample Vol., c.f. <u>84.978</u>	Sample Vol., c.f. <u>84.978</u>
	1415	1.66	1.0	216.134	295	101	91	290	42	-2	Stack Press., lwg <u>-0.81</u>	Stack Press., lwg <u>-0.81</u>
3	1420	1.67	1.0	218.846	297	101	92	293	42	-2	ΔH , lwg <u>0.8944</u>	ΔH , lwg <u>0.8944</u>
	1425	1.67	1.0	221.763	297	102	95	292	42	-2	ΔP , lwg <u>1.2173 (RM 1.4818)</u>	ΔP , lwg <u>1.2173 (RM 1.4818)</u>
2	1430	1.52	0.91	224.491	297	104	99	293	41	-2	Meter Temp., °F <u>97.83</u>	Meter Temp., °F <u>97.83</u>
	1435	1.52	0.91	227.223	297	104	101	292	40	-2	Stack Temp., °F <u>247.06</u>	Stack Temp., °F <u>247.06</u>
1	1440	1.28	0.77	229.921	297	105	101	291	39	-2	Water Collected, g <u>102.7</u>	Water Collected, g <u>102.7</u>
	1445	1.28	0.77	232.674	297	106	101	290	38	-2	O ₂ /CO ₂ S.O.T. <u>14.54</u>	O ₂ /CO ₂ S.O.T. <u>14.54</u>
STOP	1450	-	-	234.991	-	-	-	-	-	-	Comments:	Comments:

SAMPLE TRAIN TEST DATA

CLIENT Air Products/Stockton (Coke) UNIT Boiler TEST NO. 1-Hg-0 METHOD Ontario-Hydro PAGE 2 OF 2
 SAMPLE LOCATION CFBC Boiler outlet (stack) TEST CONDITION Full AMB. TEMP., °F 75 PROJECT # 99057
 OPERATOR/ASSISTANT PS/EM METER VOL. (START/END) - / - DATE 10-20-99

PRE-TEST DATA:

Barometric Press., in. Hg. _____
 Assumed Stack Press. _____
 Assumed Moisture _____
 Assumed Molecular Wt. _____
 Assumed ΔP _____
 Assumed ΔH _____
 Stack Diameter, in. _____
 Sample Time: Total _____
 per point _____
 Total of Traverse Points _____

$\Delta H =$ _____ x ΔP

EQUIPMENT INFO:

Meter No. _____
 Meter, Yd. _____
 CFM @ $\Delta H = 1.0$ _____
 Pilot ID, Cp _____
 O₂/CO₂ Method _____
 Teflon Connecting _____
 Probe: _____
 Length _____
 Nozzle _____
 Diam. _____
 Filter: _____
 No. _____
 Mat'l _____

Imp. Mat'l _____
 #16 100% 100% 100% _____
 #27 100% 100% 100% _____
 #88 100% 100% 100% _____
 #4 _____
 #5 _____
 Total _____

WL(Start) _____
 WL(End) _____
 WL(Gr) _____
 #16 687.1 _____
 #27 609.9 _____
 #88 797.7 _____
 #4 _____
 #5 _____
 Total _____

SAMPLE TRAIN LEAK CHECK:
 Pre-Test _____
 Post-Test _____

SEM Vac. _____
 Pilot _____
 Init. _____

PRE-TEST CALIBRATION CHECK:

Time _____
 Meter Reading _____
 Meter Temp. _____
 In _____
 Out _____

POST TEST INFO:

Filter Appearance _____
 Impinger Appearance _____
 Silica Gel Spent (Y/N) _____

SAMPLE POINT	TIME	METER CONDITIONS				TEMPERATURES, °F				Line Temp. °F	STATIC PRESS. iwg	CHAIN OF CUSTODY INFORMATION	
		ΔP	ΔH	METER READING	STACK	PROBE	METER IN	METER OUT	OVEN	IMP. OUT	VAC.		
1	1520	1.01	0.61	235.089	297	299	104	103	298	41	300	Impingers Loaded	
	1525	1.01	0.61	237.285	297	297	105	102	299	41	301	Impingers Recovered	
2	1530	1.31	0.79	239.647	297	297	105	102	300	40	301	Filter Loaded	
	1535	1.21	0.73	242.133	296	298	105	101	301	40	298	Filter Recovered	
3	1540	1.38	0.83	244.538	297	299	106	101	300	39	299	Probe Wash	
	1545	1.38	0.83	247.216	299	298	107	101	301	39	298	TEST SUMMARY	
4	1550	1.52	0.91	249.782	301	299	107	101	300	38	299	Calculated by:	
	1555	1.52	0.91	252.547	301	298	107	102	301	39	299	Checked by:	
5	1600	1.69	1.01	255.247	301	299	106	101	302	40	301	Sample Vol., c.f.	
	1605	1.64	0.98	258.010	301	297	104	100	302	40	300	Stack Press., iwg	
6	1610	1.64	0.98	260.846	300	299	106	101	301	41	302	ΔH , iwg	
	1615	1.75	1.05	263.796	301	300	107	101	300	41	301	ΔP , iwg	
7	1620	1.76	1.06	266.372	299	298	107	101	301	41	302	Meter Temp., °F	
	1625	1.76	1.06	269.126	299	297	107	100	300	40	301	Stack Temp., °F	
8	1630	1.69	1.01	272.214	299	298	106	99	298	41	300	Water Collected, g	
	1635	1.75	1.05	274.963	299	297	105	99	297	41	301	O ₂ /CO ₂	
STOP	1640			277.745								Comments:	

SAMPLE TRAIN TEST DATA

CLIENT: Air Products / Stockton Cogeneration UNIT: Boiler TEST NO. 2-Hg-0 METHOD: O-H PAGE: 1 OF 2
 SAMPLE LOCATION: Stockton / Boiler Outlet Bets TEST CONDITION: Full AMB. TEMP.: 65 PROJECT #: 99057
 OPERATOR/ASSISTANT: Peter Gates / EMM METER VOL. (START/END): 384.776 / 369.130 DATE: 10-21-99

PRE-TEST DATA:
 Barometric Press., in. Hg. 29.90
 Assumed Slack Press. -0.6
 Assumed Moisture 5.5
 Assumed Molecular Wt. 29.85
 Assumed ΔP 1.3
 Assumed ΔH 0.9
 Slack Distance, ft. 59.4
 Sample Time: Total 16.0
 per point 10
 Total of Traverse Points 16

$\Delta H = 0.6 \times \Delta P$

EQUIPMENT INFO:
 Meter No. N-3
 Meter, Yd. 0.996
 CFM @ ΔH 1.786
 Pilot ID, Cp 106
 O₂/CO₂ Method CEM
 Teflon Connecting Y
 Line (Y/N) Y
 Probe: Glass
 Length 10"
 Mat'l Glass
 Nozzle: 171"
 Diam. N/A
 No. Quartz
 Filter: Mat'l

Imp. Mat'l Wt. (End) Wt. (Start) Wt. (Q)
 #1 100 695.2 634.1 = 61.1
 #2 " 652.0 634.6 = 18.2
 #3 " 623.6 620.4 = 3.2
 #4 100 633.9 623.0 = 1.9
 #5 100 633.1 633.0 = 0.1
 Total (continued) 7
 POST TEST INFO: 64.6 Gray
 Filter Appearance Clear
 Impinger Appearance Clear
 Silica Gel Spent (Y/N) No

SAMPLE TRAIN LEAK CHECK:
 CEM Vac. Pilot Init.
 Pre-Test 0.009 -17 OK
 Post-Test 0.010 -16 OK

PRE-TEST CALIBRATION CHECK:
 Meter Reading In Out
 Time ΔH Meter Temp.
 Init. Final

SAMPLE POINT	TIME	METER CONDITIONS				TEMPERATURES, °F				Line Temp. °F	STATIC PRESS. lwg	CHAIN OF CUSTODY INFORMATION
		ΔP	ΔH	METER READING	PROBE	METER IN	METER OUT	OVEN	IMP. OUT			
South 8	1025	1.75	1.05	284.776	291	65	65	295	39	289	-3	Impingers Loaded
	1030	1.75	1.05	287.146	291	68	65	293	39	290	-3	Impingers Recovered
7	1035	1.85	1.11	290.007	292	75	66	294	40	290	-3	Filter Loaded
	1040	1.88	1.13	292.566	293	80	67	293	41	291	-3	Filter Recovered
6	1045	1.88	1.13	295.227	295	81	68	294	42	290	-3	Probe Wash
	1050	1.76	1.06	298.641	297	83	69	294	42	291	-3	TEST SUMMARY
5	1055	1.67	1.00	301.628	299	85	70	293	44	290	-2	Calculated by: EMM
	1100	1.67	1.00	304.892	299	87	72	294	44	290	-2	Checked by: HJC
4	1105	1.52	0.91	307.826	296	89	73	295	43	290	-2	Sample Vol., c.f. 84.254 ✓
	1110	1.52	0.91	309.543	295	89	74	296	43	290	-2	Stack Press., lwg -0.90 ✓
3	1115	1.43	0.86	312.126	294	90	75	296	42	290	-2	ΔH , lwg -0.894 ✓
	1120	1.43	0.86	314.729	294	90	76	295	42	292	-2	ΔP , lwg 1.4723 ✓
2	1125	1.15	0.69	317.344	293	92	77	296	42	291	-1	Meter Temp., °F 87.7 ✓
	1130	1.15	0.69	320.012	292	90	79	296	43	290	-1	Stack Temp., °F 294.7 ✓
1	1135	0.94	0.56	322.369	292	90	80	296	44	291	-1	Water Collected, g 100.4 ✓
	1140	0.94	0.56	324.362	294	90	81	295	45	290	-1	O ₂ /CO ₂ S.22 / 14.51 ✓
stop	1145	-	-	326.321	-	-	-	-	-	-	-	Comments:

SAMPLE TRAIN TEST DATA

CLIENT AP Stockton (Cogen) UNIT CFBC Boiler TEST NO. 2-Hg-0 METHOD OH PAGE 2 OF 2SAMPLE LOCATION Stack (outlet) TEST CONDITION Full AMB. TEMP., °F 65 PROJECT # 99057OPERATOR/ASSISTANT RG (Em) METER VOL. (START/END) - / - DATE 10-21-99

PRE-TEST DATA:

Barometric Press., in. Hg. _____

Assumed Slack Press. _____

Assumed Moisture _____

Assumed Molecular Wt. _____

Assumed ΔP _____Assumed ΔH _____

Stack Diameter, in. _____

Sample Time: Total _____

per point _____

Total of Traverse Points _____

 $\Delta H =$ _____ $\times \Delta P$

EQUIPMENT INFO:

Meter No. _____

Meter, Yd. _____

CFM @ $\Delta H = 1.0$ _____

Pilot ID, Cp _____

O₂/CO₂ Method _____

Teflon Connecting _____

Line (Y/N) _____

Mat'l _____

Length _____

Nozzle: Mat'l _____

Diam. _____

Filter: No. _____

Mat'l _____

Imp. _____

Mat'l _____

Wt (Start) _____

Wt (End) _____

Wt (Slack) _____

Wt (Q) _____

#1 _____

#2 _____

#3 _____

#4 _____

#5 _____

Total _____

POST TEST INFO:

Filter Appearance _____

Impinger Appearance _____

Silica Gel Spent (Y/N) _____

SAMPLE TRAIN LEAK CHECK:

CFM _____ Vac. _____ Init. _____

Pre-Test _____

Post-Test _____

0.010 -10 OK

PRE-TEST CALIBRATION CHECK:

Meter _____ Meter Temp. _____

Time ΔH _____ In _____ Out _____

Init. _____

Final _____

SAMPLE POINT	TIME	METER CONDITIONS			TEMPERATURES, °F							STATIC PRESS. Iwg	CHAIN OF CUSTODY INFORMATION
		ΔP	ΔH	METER READING	STACK	PROBE	METER		OVEN	IMP. OUT	VAC.		
West 8	1205	1.25	0.75	326.421	293	299	86	85	290	45	-2	Impingers Loaded	
	1210	1.30	0.78	329.891	294	298	92	86	290	45	-2		Impingers Recovered
7	1215	1.78	1.07	332.012	293	297	96	87	291	44	-2	ICE	Filter Loaded
	1220	1.78	1.07	334.166	294	296	98	88	289	45	-2		Filter Recovered
6	1225	1.01	0.61	336.948	294	297	99	91	290	45	-1		Probe Wash
	1230	1.01	0.61	339.724	294	296	98	92	291	44	-1		TEST SUMMARY
5	1235	1.25	0.75	341.932	295	296	99	92	291	44	-1		Calculated by:
	1240	1.25	0.75	344.457	295	296	101	93	290	45	-1		Checked by:
4	1245	1.75	1.05	346.901	294	295	102	93	289	45	-3	-87	Sample Vol., c.f.
	1250	1.81	1.09	349.963	295	296	104	94	288	44	-3		Stack Press., Iwg
3	1255	1.85	1.11	352.597	295	296	103	94	289	43	-3		ΔH, Iwg
	1300	1.85	1.11	356.464	294	296	108	97	290	42	-3		ΔP, Iwg
2	1305	1.51	0.91	358.697	293	295	106	97	291	41	-2		Meter Temp., °F
	1310	1.51	0.91	361.553	293	296	110	98	291	40	-2		Stack Temp., °F
1	1315	1.22	0.73	364.216	295	297	111	100	292	41	-1		Water Collected, g
	1320	1.22	0.73	366.517	294	296	112	101	291	41	-1		O ₂ /CO ₂
STOP	1325			369.130									Comments:

PRE-TEST DATA:						EQUIPMENT INFO:					
Barometric Press., in. Hg.	29.80	Meter No.	N-3	CFM @ ΔH	W-2						
Assumed Stack Press.	- .8	Pilot ID, Cp	0.996	O ₂ /CO ₂ Method							
Assumed Moisture	5.5	Teflon Connecting	1.786	Line (Y/N)							
Assumed Molecular Wt.	29.85		0.08, 0.84	Probe:	Length						
Assumed ΔP	1.2		CPM	Nozzle:	Diam.						
Assumed ΔH	0.9			Filter:	No.						
Stack Diameter - in. sq. ft.	50.27		Y		Mat'l						
Sample Time: Total	160		Glass								
per point	10		10								
Total of Traverse Points	16		Glass								
			.17"								
			Quartz								
		$\Delta H = 0.6 \times \Delta P$									

SAMPLE TRAIN LEAK CHECK:					
QEM	Yac.	Eilat	Init.		
Pre-Test	0.011	-19	OK		
Post-Test	0.010	-17	OK		
PRE-TEST CALIBRATION CHECK:					
Meter	Reading	In	Out	Meter Temp.	
Time	ΔH				
Initial					
Final					

Impo.	Matl	WL(End)	WL(Start)	WL(Q)
#1	KCl	705.1	651.6	= 53.5
#2	"	646.0	613.8	= 32.2
#3	"	637.6	632.9	= 4.7
#4	HNO ₃	621.0	621.3	= 2.7
#5	KMnO ₄	632.0	632.7	= -0.7
Total			(continued) →	
POST TEST INFO:				
Filter Appearance	Lt. Grey			
Impinger Appearance	Clear			
Silica Gel Spent (Y/N)	No			

[illegible]

9-0 METHOD 011
AMB. TEMP., °F 55
/ -

PAGE 2 OF 2
PROJECT # 99057
DATE 10/23/99

$$\Delta H = \frac{\Delta P}{x}$$

Filter: No. 11-11

Impo.	Mat'l	WL(End)	WL(Start)	WL(G)
6.44 gm	kmno ₄ /H ₂ SO ₄	646.7	647.2	-0.5
7.44	"	603.3	602.9	0.4
8.48	5.5	821.8	807.1	14.7
#4				
#5				
Total				

POST TEST INFO:
 Filter Appearance
 Impinger Appearance
 Silica Gel Spent (Y/N)

See p. 1

	<u>CFM</u>	<u>Vac.</u>	<u>Pitot</u>	<u>Inlet</u>
1				
2				
3				
4				
5				
6				
7				
8				
9				
10				
11				
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96				
97				
98				
99				
100				

Pre-Test

Post-Test

PRE-TEST CALIBRATION CHECK:

Time	ΔH	Reading	Meter Temp.
		In	Out

init

[illegible]

SAMPLE TRAIN TEST DATA

CLIENT Air Products UNIT Bayshore In TEST NO. 1-14-Ink METHOD Ontario Hydro PAGE 1 OF 3
 SAMPLE LOCATION Stack 3A TEST CONDITION Full AMB. TEMP., °F 74.143 PROJECT # 99057
 OPERATOR/ASSISTANT JP/KSC/EM/DOJ METER VOL. (START/END) 682422 DATE 10.20.99

PRE-TEST DATA:

Barometric Press., in. Hg. 29.95
 Assumed Stack Press. 12
 Assumed Moisture 6.0
 Assumed Molecular Wt. 29.85
 Assumed ΔP 0.98
 Assumed ΔH 0.33
 Stack Diameter, in. 85.7110
 Sample Time: Total 1408
 per point 24
 Total of Traverse Points 24

$$\Delta H = 0.40 \times \Delta P$$

EQUIPMENT INFO:

Meter No. N-2
 Meter, Yd. 1.018
 CFM @ $\Delta H = 1.0$ 1.756
 Pilot ID, Cp 90.9
 O₂/CO₂ Method 32
 Teflon Connecting Y
 Line (Y/N) Y
 Probe: Pyrex
 Length 81
 Mat'l Pyrex
 Nozzle: Pyrex
 Diam. 1/4
 No. 1
 Filter: 1/4
 Mat'l Pyrex

Imp. #1 100ml 500ml WL(End) 635.8 WL(Start) 584.5 WL(a) 41.3
#2 " " 641.7 629.0 12.7
#3 " " 630.3 628.2 2.0
#4 100ml 500ml 620.3 619.1 1.2
#5 100ml 500ml 627.8 624.4 4.67
 Total 397.155"
 POST TEST INFO: (on page 2) ash
 Filler Appearance load 1/2 grey ash
 Impinger Appearance Clear
 Silica Gel Spent (Y/N) No

SAMPLE TRAIN LEAK CHECK:

CEM Vac. Pilot Init.
 Pre-Test 0.015 22" ve 1/2"
 Post-Test 0.006 18" ve 1/2"

PRE-TEST CALIBRATION CHECK:

Meter Meter Temp.
 Time ΔH Reading In Out
 Init. ---
 Final ---

SAMPLE POINT	TIME	METER CONDITIONS				TEMPERATURES, °F				SL. Temp. °F	STATIC PRESS. iwg	CHAIN OF CUSTODY INFORMATION
		ΔP	ΔH	METER READING	PROBE	METER IN	METER OUT	OVEN	IMP. OUT			
A-3	1324	0.83	0.33	682.422	300	892	94	94	58	304	4	Impingers Loaded DD
	1328	0.85	0.34	683.520	301	891	95	94	57		4	Impingers Recovered DD
A-2	1330	0.87	0.35	684.640	301	890	96	95	56		4	Filter Loaded DD
	1335	0.88	0.35	686.135	301	894	96	95	56		4	Filter Recovered DD
A-1	1338	0.97	0.45	687.120	301	897	96	95	56		4	Probe Wash JP/KSC
	1342	0.93	0.37	688.580	300	898	96	95	57	307	4	TEST SUMMARY
A-6	1345	1.1	0.44	689.660	300	895	97	95	58		5	Calculated by: EM
	1349	1.1	0.44	691.020	301	894	98	95	59		5	Checked by: KJC
A-5	1352	1.1	0.44	692.130	301	895	98	95	59		5	Sample Vol., c.f. 57.221 ✓
	1356	1.1	0.44	693.620	301	897	99	95	59		5	Stack Press., iwg -12.4 ✓
A-4	1359	0.80	0.32	694.670	301	896	99	95	59		5	ΔH , iwg 0.395 ✓
	1403	0.78	0.31	696.080	301	897	99	95	59		4	ΔP , iwg 0.9811 ✓
	1406			696.940								Meter Temp., °F 99.8 ✓
				(14.518)								Stack Temp., °F 301.5 ✓
B-6	1422	1.0	0.40	698.743	304	893	95	95	58	308	5	Water Collected, g 73.3 ✓
	1426	1.1	0.44	700.820	305	895	99	95	60	310	5	O ₂ /CO ₂ 3.70 / 15.82 ✓
B-5	1429	1.1	0.44	701.220	305	896	99	95	58	309	5	Comments: S.L. Ruse to
	1433	1.1	0.44	702.500	305	893	100	96	57	310	5	#1 100ml 500ml 100g 0.1N H ₂ O ₂

SAMPLE TRAIN TEST DATA

CLIENT Air Products UNIT Bryant Inlet TEST NO. 1-Hy-Inlet METHOD OH PAGE 2 OF 3
 SAMPLE LOCATION Stockton, CA TEST CONDITION Full AMB. TEMP., °F - PROJECT # 991057
 OPERATOR/ASSISTANT JR/KS/EM/OZO METER VOL. (START/END) - / - DATE 10-20-99

PRE-TEST DATA:

Barometric Press., in. Hg. 30.1
 Assumed Slack Press. 0.0
 Assumed Moisture 0.0
 Assumed Molecular Wt. 16.0
 Assumed ΔP 0.0
 Assumed ΔH 0.0
 Slack Diameter, in. 0.40
 Sample Time: Total per point 0.40 x ΔP
 Total of Traverse Points 1

EQUIPMENT INFO:

Meter No. 12-2
 Meter, Yd. 1.018
 CFM @ $\Delta H = 1.0$ 1.756
 Pilot ID, Cp 910-1 0.54
 O₂/CO₂ Method 3P
 Teflon Connecting Y
 Line (Y/N) Y
 Probe: Mat'l Al
 Length 3.9
 Nozzle: Mat'l Al
 Filter: No. 10.0mm 0.5
 Mat'l Al

Imp.	Mat'l	WL(End)	WL(Start)	WL(Q)
#16	100.05	634.7	635.1	634.7
#27	"	640.7	640.1	640.6
#38	5.6el	607.8	790.3	17.5
#4				
#5				
Total				73.3

POST TEST INFO:
 Filter Appearance See 7.1
 Impinger Appearance See 7.1
 Silica Gel Spent (Y/N) See 7.1

SAMPLE TRAIN LEAK CHECK:

Pre-Test CEM Vac. Pilot Init.
 Post-Test CEM Vac. Pilot Init.

PRE-TEST CALIBRATION CHECK:

Time 2.7 Meter Reading 2.7 Meter Temp. 2.7
 Init. In Out
 Final In Out

SAMPLE POINT	TIME	METER CONDITIONS			TEMPERATURES, °F							STATIC PRESS. iwg	CHAIN OF CUSTODY INFORMATION
		ΔP	ΔH	METER READING	STACK	PROBE	METER		OVEN	IMP. OUT			
							IN	OUT					
B-4	1436	0.99	0.40	703.840	303	295	102	96	292	55	312	5	Impingers Loaded
	1440	0.97	0.39	705.240	303	293	100	96	290	50	313	5	Impingers Recovered
B-3	1443	0.80	0.34	706.330	302	293	100	96	293	49	313	5	Filter Loaded
	1447	0.86	0.34	707.560	302	295	101	97	294	49	314	5	Filter Recovered
B-2	1450	0.84	0.34	708.465	302	297	101	97	295	48	314	5	Probe Wash
	1454	0.85	0.34	709.040	301	299	102	98	297	49	315	5	TEST SUMMARY
B-1	1457	0.94	0.36	710.580	301	300	102	97	298	48	315	5	Calculated by:
	1500	0.94	0.36	711.450	300	298	102	98	295	48	315	5	Checked by:
END	1504			712.658 (13.915)									Sample Vol., c.f.
													Stack Press., iwg
													ΔH, iwg
C-6	1506	1.1	0.44	713.715	301	305	103	98	296	50	316	5	ΔP, iwg
	1510	1.1	0.44	714.790	301	305	103	98	296	49	318	5	Meter Temp., °F
C-5	1513	1.1	0.44	715.900	302	303	103	98	296	49	318	5	Stack Temp., °F
	1517	1.1	0.44	717.370	298	302	104	98	298	49	318	5	Water Collected, g
C-4	1520	0.93	0.37	718.460	302	290	104	98	298	50	318	5	O ₂ /CO ₂
	1524	0.93	0.37	719.800	302	291	104	99	298	51	318	5	Comments:
C-3	1527	0.85	0.34	720.820	301	292	104	99	297	52	318	5	

SAMPLE TRAIN TEST DATA

CLIENT Air Products UNIT Bayview Inlet TEST NO. 1-Hy-Inlet METHOD OH PAGE 3 OF 3
 SAMPLE LOCATION Stockton, CA TEST CONDITION Full AMB. TEMP., °F 85 PROJECT # 99057
 OPERATOR/ASSISTANT JL/LWC/BJO/Km METER VOL. (START/END) / DATE 10-20-99

PRE-TEST DATA:

Barometric Press., in. Hg. _____
 Assumed Stack Press. _____
 Assumed Moisture _____
 Assumed Molecular Wt. _____
 Assumed ΔP _____
 Assumed ΔH _____
 Stack Diameter, in. _____
 Sample Time: Total _____ per point
 Total of Traverse Points _____

$$\Delta H = 0.40 \times \Delta P$$

EQUIPMENT INFO:

Meter No. N-2
 Meter, Yd. 1-018
 CFM @ $\Delta H = 1.0$ 1-756
 Pilot ID, Cp 910-0054
 O₂/CO₂ Method 32
 Teflon Connecting _____
 Line (Y/N) _____
 Probe: Mat'l _____ Length _____
 Nozzle: Mat'l _____ Diam. _____
 Filter: No. _____
 Mat'l _____

IMP. Mat'l Wt.(End) Wt.(Start) Wt.(Lo)

#1 _____
 #2 _____
 #3 _____
 #4 _____
 #5 _____
 Total _____
 POST TEST INFO:
 Filter Appearance _____
 Impinger Appearance _____
 Silica Gel Spent (Y/N) _____

SAMPLE TRAIN LEAK CHECK:

CFM Vac. Pilot Init.
 Pre-Test _____
 Post-Test _____

PRE-TEST CALIBRATION CHECK:

Time _____ Meter Reading _____ Meter Temp. _____
 In _____ Out _____
 Init. _____
 Final _____

SAMPLE POINT	TIME	METER CONDITIONS			TEMPERATURES, °F						STATIC PRESS. iwg	CHAIN OF CUSTODY INFORMATION	
		Δ P	Δ H	METER READING	STACK	PROBE	METER		OVEN	IMP. OUT			
							IN	OUT			VAC.		
C-3	1531	0.85	0.34	702.190	301	092	103	09	096	53	318	5	Impingers Loaded
C-2	1534	0.90	0.36	703.145	301	091	104	09	097	53	318	5	Impingers Recovered
	1538	0.88	0.35	704.480	301	090	104	100	098	52	318	5	Filter Loaded
C-1	1541	1.0	0.40	705.490	300	090	104	100	095	51	319	5	Filter Recovered
	1545	1.0	0.40	706.780	300	088	103	100	093	52	319	5	Probe Wash
END	1548			707.578									TEST SUMMARY
				(17.163)									Calculated by:
D-6	1552	1.3	0.52	709.518	300	085	105	100	094	52	320	6	Checked by:
	1558	1.2	0.48	731.865	300	087	106	101	094	54	321	6	Sample Vol., c.f.
D-5	1559	0.90	0.36	732.320	300	085	106	101	095	55	321	6	Stack Press., iwg
	1603	0.95	0.38	733.750	300	086	106	101	094	55	321	6	Δ H, iwg 0.40
D-4	1606	1.0	0.40	734.720	300	084	106	101	092	55	321	6	Δ P, iwg 0.9905
	1610	1.1	0.44	736.050	303	080	106	102	091	52	321	6	Meter Temp., °F 0.9
D-3	1613	1.1	0.44	737.140	304	082	107	102	090	50	321	6	Stack Temp., °F
	1617	1.1	0.44	738.720	303	080	107	102	091	49	321	6	Water Collected, g
D-2	1620	1.0	0.40	739.600	304	081	107	102	090	49	321	6	O ₂ /CO ₂
	1625	1.0	0.40	741.410	302	080	107	103	090	49	321	6	Comments:
D-1	1627	1.0	0.40	742.180	301	081	107	102	091	49	321	6	END 16.34 744.143

SAMPLE TRAIN TEST DATA

CLIENT Air Products UNIT Bayshore Inlet TEST NO. 2-Hg-Inlet METHOD Ontario Hydro PAGE 1 OF 3
SAMPLE LOCATION Stack 1, CA 05 TEST CONDITION Full AMB. TEMP., °F 73 PROJECT # 99057
OPERATOR/ASSISTANT JP/KVC/DW/EM METER VOL. (START/END) 752.252 / 82.796 DATE 10-21-99

PRE-TEST DATA:
Barometric Press., in. Hg. 29.90
Assumed Stack Press. 29.85
Assumed Moisture 5.9
Assumed Molecular Wt. -12.0
Assumed ΔP 0.39
Assumed ΔH 0.39
Stack Diameter, in. 24 x 110
Sample Time: Total 168
per point 7
Total of Traverse Points 24
 $\Delta H = 0.40 \times \Delta P$

EQUIPMENT INFO:
Meter No. N-2
Meter, Yd. 1.015
CFM @ $\Delta H = 1.0$ 1.716
Pilot ID, Cp 91.0084
O₂/CO₂ Method 3a
Teflon Connecting Y
Line (Y/N) Y
Matl' Pvc
Length 91'
Probe: Matl' Pvc
Nozzle: Matl' 3.9 / 1.55"
Filter: No. 90mm 24x
Matl' Clear

IMP. Matl' WL(End) WL(Start) WL(Q) VAC.
#1 100 nls KCI 631.0 581.2 49.8
#2 " 636.6 608.6 8.0
#3 " 637.1 627.2 -0.1
#4 100 nls H₂O 633.3 621.6 1.7
#5 100 nls KNO₃/H₂O 630.7 620.4 0.3
Total (Cont. On Pg 2) →
POST TEST INFO: loaded w/ grey Ash
Filler Appearance Clear
Impinger Appearance Clear
Silica Gel Spent (Y/N) Y

SAMPLE TRAIN LEAK CHECK:
CEM Vac. Pilot Init.
Pre-Test .005 17" OK EM
Post-Test .009 12" OK EM
PRE-TEST CALIBRATION CHECK:
Meter Meter Temp.
Time ΔH Reading In Out
Init. — — — —
Final — — — —

SAMPLE POINT	TIME	METER CONDITIONS				TEMPERATURES, °F				STATIC PRESS. iwg	CHAIN OF CUSTODY INFORMATION	
		ΔP	ΔH	METER READING	STACK	PROBE	METER IN	METER OUT	OVEN	IMP. OUT	VAC.	
D-6	1012	0.83	0.33	750.252	2910	306	77	77	305	60	4	Impingers Loaded <u>DP</u>
	1016	1.1	0.44	753.600	2913	303	80	75	304	58	4	Impingers Recovered <u>DP/PG</u>
D-5	1019	0.93	0.37	754.460	2913	303	82	76	300	58	5	Filter Loaded <u>DP</u>
	1023	0.90	0.36	756.035	2913	302	84	77	301	57	4	Filter Recovered <u>DP</u>
D-4	1026	1.1	0.44	757.020	2914	302	85	78	300	57	5	Probe Wash <u>1P/1KSC</u>
	1030	1.1	0.44	758.425	2915	304	85	78	302	55	5	TEST SUMMARY
D-3	1033	1.1	0.44	759.530	2916	299	87	80	300	53	5	Calculated by: <u>EM</u>
	1037	1.1	0.44	760.990	2918	295	88	81	301	51	5	Checked by: <u>KJC</u>
D-2	1040	0.97	0.39	762.120	2919	299	89	82	299	51	5	Sample Vol., c.f. <u>57.706</u> ✓
	1044	0.97	0.39	763.470	2919	289	90	82	295	51	5	Stack Press., iwg <u>-12.0</u> ✓
D-1	1047	1.0	0.40	764.470	2918	288	89	83	292	50	5	ΔH , iwg <u>0.376</u> ✓
	1051	0.97	0.39	765.860	2918	289	90	83	290	50	5	ΔP , iwg <u>0.9367</u> ^{RMS}
END	1054			766.533								Meter Temp., °F <u>94.1</u> ✓
												Stack Temp., °F <u>298.2</u> ✓
C-6	1056	1.0	0.40	767.825	3010	303	92	85	295	48	5	Water Collected, g <u>76.2</u> ✓
	1100	1.0	0.40	769.180	3013	300	92	85	292	49	5	O ₂ /CO ₂ <u>4.36 / 14.99</u> ✓
C-5	1103	0.96	0.38	770.200	301	299	92	86	294	50	5	Comments:
	1107	0.95	0.38	771.710	2919	297	93	86	292	49	5	

SAMPLE TRAIN TEST DATA

PAGE 2 OF 3

UNIT Baghouse Inlet TEST NO. 2-H₂-Inlet METHOD OH

PROJECT #

AMB. TEMP., °F

TEST CONDITION

CLIENT Air ProductsDATE 10-31-99

METER VOL. (START/END)

SAMPLE LOCATION Stack 2, 2A

METER VOL. (START/END)

OPERATOR/ASSISTANT JL/KJC/PCG/EN

PRE-TEST DATA:

Barometric Press., in. Hg.

Assumed Slack Press.

Assumed Moisture

Assumed Molecular Wt.

Assumed ΔP Assumed ΔH

Stack Diameter, in.

Sample Time: Total

per point

Total of Traverse Points

 $\Delta H = 0.40 \times \Delta P$

EQUIPMENT INFO:

Meter No.

Meter, Yd.

CFM @ $\Delta H = 1.0$

Pilot ID, Cp

O₂/CO₂ Method

Teflon Connecting

Line (V/N)

Probe:

Length

Nozzle:

Diam.

Filter:

No.

Mat'l

Imp. Mat'l Wt. (Start) Wt. (End)

6 N 100m lb 632.4 632.5

7 11 643.1 644.1

8 5.6 840.8 843.2

#4

#5

Total

POST TEST INFO:

Filter Appearance

Impinger Appearance

Silica Gel Spent (V/N)

See P. 1

SAMPLE TRAIN LEAK CHECK:

CEM Vac. Pilot Init.

Pre-Test

Post-Test

PRE-TEST CALIBRATION CHECK:

Meter

Meter Temp.

Time

Readings

Init.

Final

SAMPLE POINT	TIME	METER CONDITIONS				TEMPERATURES, °F				IMP. OUT	OVEN	VAC.	STATIC PRESS. iwg	CHAIN OF CUSTODY INFORMATION
		ΔP	ΔH	METER READING	STACK	PROBE	METER IN	METER OUT	TEMP °F					
C-4	1110	0.85	0.34	772.655	097	090	93	87	323	49	095	5		Impingers Loaded
	1114	0.85	0.34	774.075	097	088	94	87	314	50	092	5		Impingers Recovered
C-3	1117	0.78	0.31	775.000	095	085	93	88	315	50	090	5		Filter Loaded
	1121	0.80	0.33	776.320	095	080	94	88	316	50	089	5		Filter Recovered
C-2	1124	0.83	0.33	777.260	096	080	94	88	316	51	088	5	-11.8	Probe Wash
	1128	0.87	0.35	778.530	096	081	95	89	317	52	087	5		TEST SUMMARY
C-1	1131	0.93	0.37	779.590	095	080	96	89	317	52	085	5		Calculated by:
	1135	0.96	0.38	780.930	095	080	97	90	317	52	085	5		Checked by:
ENO	1138			781.777										Sample Vol., c.f.
														Stack Press., iwg
B-6	1140	1.0	0.40	782.638	300	300	98	91	318	51	096	5		ΔH , iwg
	1144	1.0	0.40	784.260	302	309	100	92	319	53	096	5		ΔP , iwg
B-5	1147	0.93	0.37	785.390	300	290	101	93	319	54	300	5		Meter Temp., °F
	1151	0.99	0.40	786.920	300	286	103	94	319	55	299	5		Stack Temp., °F
B-4	1154	0.94	0.38	788.030	300	285	104	95	319	55	299	5		Water Collected, g
	1158	0.95	0.38	789.445	299	282	104	95	319	55	292	5		O ₂ /CO ₂
B-3	1201	0.85	0.34	790.450	299	280	104	96	319	55	290	5		Comments:
	1205	0.88	0.35	791.630	299	279	104	97	319	55	289	5		

SAMPLE TRAIN TEST DATA

CLIENT Arc Products UNIT Booths Inlet TEST NO. 2-Hy-Inlet METHOD OH PAGE 3 OF 3
 SAMPLE LOCATION Stacks, CA TEST CONDITION Full AMB. TEMP., °F - PROJECT # 99057
 OPERATOR/ASSISTANT JP/KLC/DA/EM METER VOL. (START/END) - DATE 10-31-99

PRE-TEST DATA:
 Barometric Press., in. Hg. _____
 Assumed Stack Press. _____
 Assumed Moisture _____
 Assumed Molecular Wt. _____
 Assumed ΔP _____
 Assumed ΔH _____
 Stack Diameter, in. _____
 Sample Time: Total _____ per point
 Total of Traverse Points _____

EQUIPMENT INFO:
 Meter No. U-2
 Meter, Yd. 1-218
 CFM @ ΔH = 1.0 1-256
 Pilot ID, Cp 9'P-2084
 O₂/CO₂ Method 3A
 Teflon Connecting _____
 Line (Y/N) _____
 Probe: Mat'l _____ Length _____
 Nozzle: Mat'l _____ Diam. _____
 Filter: No. _____ Mat'l _____

POST TEST INFO:
 Filter Appearance _____
 Impinger Appearance _____
 Silica Gel Spent (Y/N) _____

SAMPLE TRAIN LEAK CHECK:
 CFM Vac. _____ Init. _____
 Pre-Test _____
 Post-Test _____
 PRE-TEST CALIBRATION CHECK:
 Meter _____
 Time _____ Reading _____
 Meter Temp. _____
 In _____ Out _____

ΔH = 6.40 x ΔP

SAMPLE POINT	TIME	METER CONDITIONS				TEMPERATURES, °F					STATIC PRESS. iwg	CHAIN OF CUSTODY INFORMATION
		ΔP	ΔH	METER READING	STACK	PROBE	METER		OVEN	IMP. OUT		
							IN	OUT				
B-2	1208	0.82	0.33	792.540	248	279	104	98	289	56	5	Impingers Loaded
	1212	0.85	0.34	794.190	299	279	104	98	290	57	5	Impingers Recovered
B-1	1215	0.90	0.36	795.200	297	278	104	99	289	56	5	Filter Loaded
	1219	0.92	0.37	796.450	298	277	105	99	287	54	5	Filter Recovered
END	1222			797.398								Probe Wash
TEST SUMMARY												
A-6	1224	1.1	0.44	798.383	301	303	106	101	296	50	6	Calculated by:
	1229	1.1	0.44	800.270	302	301	107	102	293	50	6	Checked by:
A-5	1231	1.1	0.44	800.470	303	300	107	101	295	49	6	Sample Vol., c.f.
	1235	1.1	0.44	802.465	303	298	106	101	297	49	6	Stack Press., iwg
A-4	1238	0.75	0.30	803.535	302	295	106	101	296	50	5	ΔH, iwg
	1242	0.75	0.30	804.810	301	290	105	102	293	50	5	ΔP, iwg
A-3	1245	0.88	0.35	805.765	300	286	105	101	290	50	5	Meter Temp., °F
	1249	0.85	0.34	807.040	300	283	107	101	290	50	5	Stack Temp., °F
A-2	1252	0.85	0.34	808.040	299	280	107	102	288	51	5	Water Collected, g
	1256	0.85	0.34	809.360	299	277	107	103	286	51	5	O ₂ /CO ₂
	1259	0.95	0.38	810.370	296	276	107	103	283	52	5	Comments:
A-1	1303	0.93	0.37	811.675	296	275	107	103	280	52	5	

SAMPLE TRAIN TEST DATA

CLIENT Stockton Cogen UNIT CEBC TEST NO. 3-Hg-I METHOD OH PAGE 1 OF 3
 SAMPLE LOCATION Bogobose Inlet TEST CONDITION Full AMB. TEMP., °F 70 PROJECT # 99057
 OPERATOR/ASSISTANT JP/KJC METER VOL. (START/END) 835.364 / 877.787 DATE 10/22/99

PRE-TEST DATA:
 Barometric Press., in. Hg. 29.80
 Assumed Slack Press. 0.008
 Assumed Moisture 0.013
 Assumed Molecular Wt. 12.0
 Assumed ΔP 0.08
 Assumed ΔH 0.013
 Stack Diameter, in. 12.0
 Sample Time: Total per point 0.013
 Total of Traverse Points 12.0

EQUIPMENT INFO:
 Meter No. N-2
 Meter, Yd. 1.018
 CFM @ ΔH = 1.0 1.756
 Pilot ID, Cp 1.256
 O₂/CO₂ Method 3.2
 Teflon Connecting Line (Y/N) Y
 Probe: Mat'l HN03/06
 Length Y
 Mat'l H₂O₂
 Nozzle: Mat'l HN03/06
 Diam. 3.4
 Filter: No. 1.155
 Mat'l 3.4

POST TEST INFO:
 Filter Appearance Loaded w/ 6 grey Ash
 Impinger Appearance Clear
 Silica Gel Spent (Y/N) No

PRE-TEST CALIBRATION CHECK:
 Meter Reading In Out
 Time ΔH Reading In Out
 Meter Temp. In Out

SAMPLE TRAIN LEAK CHECK:
 CFM Vac. Pilot Init.
 Pre-Test 0.008 15 16.5 EM
 Post-Test 0.013 12 12 EM

SAMPLE POINT	TIME	METER CONDITIONS				TEMPERATURES, °F				STATIC PRESS. iwg	CHAIN OF CUSTODY INFORMATION	
		ΔP	ΔH	METER READING	PROBE	METER IN	METER OUT	OVEN	IMP. OUT			
A-6	0850	1.1	0.44	835.264	296	63	62	292	45	5	Impingers Loaded	DD/EM
	0854	1.1	0.44	836.730	298	65	63	295	45	5	Impingers Recovered	DD
5	0857	1.2	0.48	837.780	300	66	64	293	45	5	Filter Loaded	DD
	0901	1.2	0.48	839.870	300	68	65	295	45	5	Filter Recovered	DD
4	0904	0.80	0.32	840.360	300	70	66	292	45	4	Probe Wash	JP/KJC
	0908	0.83	0.33	841.590	300	71	66	290	45	4	TEST SUMMARY	
3	0911	0.93	0.37	842.520	300	73	67	288	45	4	Calculated by:	EM
	0915	0.95	0.38	843.795	299	74	68	285	45	4	Checked by:	KJC
2	0918	0.91	0.36	844.830	299	75	68	284	45	4	Sample Vol., c.f.	57.783 ✓
	0922	0.90	0.36	846.185	299	76	69	284	45	5	Stack Press., iwg	-12.1 ✓
1	0925	0.96	0.38	847.230	296	77	70	281	45	5	ΔH, iwg	0.395 ✓
	0929	0.97	0.39	848.580	294	77	71	283	45	5	ΔP, iwg	0.9876 ✓
END	0932			849.482							Meter Temp., °F	88.81.7 ✓
											Stack Temp., °F	296.7 ✓
B-6	0933	1.1	0.44	850.028	300	79	72	290	45	5	Water Collected, g	76.9 ✓
	0937	1.1	0.44	851.500	301	80	73	288	46	5	O ₂ /CO ₂ 3.87	15.76 ✓
B-5	0940	1.0	0.40	852.495	300	80	73	287	47	5	Comments:	
	0944	0.98	0.39	853.870	300	81	74	288	46	5		

SAMPLE TRAIN TEST DATA

CLIENT Air Products/Stockton Cogeneration UNIT Bag-In TEST NO. 3-Hg-I METHOD OH PAGE 2 OF 3
 SAMPLE LOCATION Stockton CA Duct TEST CONDITION Full AMB. TEMP., °F - PROJECT # 90057
 OPERATOR/ASSISTANT JP/KJC METER VOL. (START/END) - DATE 10-22-94

PRE-TEST DATA:

Barometric Press., In. Hg. 30.1
 Assumed Stack Press. 0.1
 Assumed Moisture -0.1
 Assumed Molecular Wt. 12.0
 Assumed ΔP -
 Assumed ΔH -
 Stack Diameter, in. 3.9
 Sample Time: Total 90 min per point
 Total of Traverse Points 4

$$\Delta H = 0.40 \times \Delta P$$

EQUIPMENT INFO:

Meter No. U-2
 Meter, Yd. 1.018
 CFM @ $\Delta H = 1.0$ 1.166
 Pilot ID, Cp. 9 P-D 085
 O₂/CO₂ Method 34
 Teflon Connecting Y
 Line (Y/N) Y
 Probe: Pyrex
 Length 91
 Nozzle: Pyrex
 Diam. 3.9
 Filter: 90 mm Q14
 No. 3.9
 Mat'l 3.9

IMP. Maltl WL(End) WL(Start) WL(Q)
 #1 KM-04/1504 638.1 638.1 0.1
 #2 " 642.6 642.7 -0.1
 #3 S.G. 831.1 819.1 12.0
 #4 - - - -
 #5 - - - -
 Total See P.1
 POST TEST INFO:
 Filter Appearance -
 Impinger Appearance -
 Silica Gel Spent (Y/N) -

SAMPLE TRAIN LEAK CHECK:
 CEM Vac. Pilot Init.

Pre-Test See
 Post-Test See

PRE-TEST CALIBRATION CHECK:

Meter Meter
 Time ΔH Reading In Out
 Init. 9.1
 Final -

SAMPLE POINT	TIME	METER CONDITIONS			TEMPERATURES, °F							STATIC PRESS. iwg	CHAIN OF CUSTODY INFORMATION
		Δ P	Δ H	METER READING	STACK	PROBE	METER		OVEN	IMP. OUT			
							IN	OUT					
B-4	0947	0.910	0.38	851.900	299	288	81	75	283	47	306	5	Impingers Loaded
B-3	0951	0.910	0.38	856.265	299	285	82	75	286	47	307	5	Impingers Recovered
	0954	0.89	0.36	857.270	298	278	83	76	285	48	307	5	Filter Loaded
B-2	0958	0.91	0.36	858.790	298	276	84	77	283	48	307	5	Filter Recovered
	1001	0.85	0.34	859.420	298	273	84	78	282	49	306	5	Probe Wash
B-1	1005	0.85	0.34	860.720	296	274	85	79	280	49	306	5	TEST SUMMARY
	1008	0.93	0.37	861.700	295	275	85	79	279	49	306	5	Calculated by:
END	1012	0.91	0.36	862.990	295	276	86	80	279	50	307	5	Checked by:
	1015			863.948									Sample Vol., c.f.
C-6	1017	1.1	0.44	864.828	295	203	87	81	283	49	307	5	Stack Press., iwg
	1021	1.1	0.44	866.370	296	202	87	81	288	49	308	5	Δ H, iwg
C-5	1024	1.1	0.44	867.490	297	209	87	81	285	49	309	5	Δ P, iwg
	1028	1.1	0.44	869.040	297	218	88	82	286	49	310	5	Meter Temp., °F
C-4	1031	0.81	0.32	870.200	297	285	89	83	287	50	310	5	Stack Temp., °F
	1035	0.90	0.36	871.450	297	283	89	83	285	51	311	5	Water Collected, g
C-3	1038	0.88	0.35	872.420	297	280	89	83	283	52	311	5	O ₂ /CO ₂
	1042	0.83	0.33	873.900	296	277	90	84	280	53	312	5	Comments:

SAMPLE TRAIN TEST DATA

CLIENT Av Products Stacker Co UNIT Bayview Int'l TEST NO. 3-Hg-I METHOD OH PAGE 3 OF 3
 SAMPLE LOCATION Stacker Co TEST CONDITION Full AMB. TEMP., °F 75 PROJECT # 99057
 OPERATOR/ASSISTANT JF/KJC METER VOL. (START/END) - METER TEMP., °F - DATE 10-22-99

PRE-TEST DATA:
 Barometric Press., in. Hg. _____
 Assumed Stack Press. _____
 Assumed Moisture _____
 Assumed Molecular Wt. _____
 Assumed ΔP _____
 Assumed ΔH _____
 Stack Diameter, in. _____
 Sample Time: Total _____ per point
 Total of Traverse Points _____

$\Delta H = 0.40 \times \Delta P$

EQUIPMENT INFO:
 Meter No. N-2
 Meter, Yd. 1.018
 CFM @ $\Delta H = 1.0$ 1.756
 Pilot ID, Cp. 9.1 P-D 0.84
 O₂/CO₂ Method 3A
 Teflon Connecting Line (Y/N) Y
 Probe: Y Mat'l _____ Length _____
 Nozzle: Y Mat'l _____ Diam. _____
 Filter: _____ No. _____ Mat'l _____

TEMPERATURES, °F

SAMPLE POINT	TIME	METER CONDITIONS		TEMPERATURES, °F				IMP. OUT	VAC.	STATIC PRESS. lwg	CHAIN OF CUSTODY INFORMATION
		ΔP	ΔH	PROBE	METER IN	METER OUT	OVEN				
C-2	1045	0.88	0.35	075	90	84	080	54	5		Impingers Loaded Impingers Recovered Filter Loaded Filter Recovered Probe Wash TEST SUMMARY Calculated by: Checked by: Sample Vol., c.f. Stack Press., iwg ΔH , iwg ΔP , iwg Meter Temp., °F Stack Temp., °F Water Collected, g O ₂ /CO ₂ Comments:
	1049	0.88	0.35	075	90	85	081	54	5		
C-1	1052	1.0	0.40	074	90	85	080	55	5		
	1056	0.99	0.40	072	91	86	081	55	5		
END	1059										
D-6	1107	1.21	0.48	275	93	87	082	54	6		
	1112	1.20	0.48	276	94	87	080	55	6		
D-5	1114	1.0	0.40	275	94	88	081	57	6		
	1118	0.98	0.39	274	94	88	080	53	6		
D-4	1121	1.1	0.44	277	95	89	080	49	6	-124	
	1125	1.1	0.44	277	96	89	082	49	6		
D-3	1128	1.1	0.44	278	96	90	083	47	6		
	1132	1.1	0.44	277	96	91	082	47	6		
D-2	1135	1.0	0.40	277	96	90	084	47	6		
	1139	1.0	0.40	278	97	91	083	47	6		
D-1	1142	0.95	0.38	277	98	92	084	48	6		
	1146	0.95	0.38	276	98	92	083	47	6		
END	1149				97	87					

POST TEST INFO:
 Filter Appearance _____
 Impinger Appearance _____
 Silica Gel Spent (Y/N) _____

SAMPLE TRAIN LEAK CHECK:
 CFM _____ Vac. _____ Init. _____
 Pre-Test _____
 Post-Test _____

PRE-TEST CALIBRATION CHECK:
 Meter _____ Meter Temp. _____
 Time ΔH _____ Reading _____ In _____ Out _____

Appendix C.5

Fuel Analysis Data



A.J. EDMOND CO.

1530 TEXAS AVENUE
TEXAS CITY, TEXAS 77590
(409) 948-4504
FAX (409) 948-4046

LABORATORIES

ANALYTICAL
CARGO SURVEYS
COMMODITY SURVEY
INSPECTORS
CONSULTANTS
MANUFACTURING
BARGE SURVEYS
SAMPLERS
SAMPLING SYSTEMS

November 18, 1999

Air Products Stockton Cogen
1010 Zephyr Street
Stockton, CA 95206

Project Number: TC00557
Sample Number: TC022135
Coal #SCCHG11099BC

Attn: Ms. Tara Keefover

LABORATORY ANALYSES

As Received Basis:

Moisture, %	6.12
-------------	------

Dry Basis:

Volatile Matter, %	38.19
Ash, %	13.07
Fixed Carbon, %	48.74
Sulfur, %	0.64
Oxygen (by difference), %	9.48
Calorific Value, Gross, BTU/Lb	12334
Chlorine, ppm	668
Mercury, ppm	0.026

Respectfully Submitted

A. J. EDMOND COMPANY

by K. J. Kumke
K. J. Kumke



A.J. EDMOND CO.

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Air Products Stockton Cogen
1010 Zephyr Street
Stockton, CA 95206

November 17, 1999

Project Number: TC00557
Sample Number: TC022135
Coal, SCCHG11099BC

Certificate Of Analysis

Ultimate Analyses

Carbon, %
Hydrogen, %
Nitrogen, %
Sulfur, %
Ash, %
Oxygen (by difference), %
Total Moisture, %

Dry Basis

70.47
4.90
1.45
0.64
13.07
9.48

As-Received


66.15
5.29
1.36
0.60
12.27
14.33
6.12

KJK/100

Respectfully Submitted
A. J. EDMOND COMPANY

Form 2.10f1

by


K. J. Kumke



A.J. EDMOND CO.

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Air Products Stockton Cogen
1010 Zephyr Street
Stockton, CA 95206

November 17, 1999

Project Number: TC00557
Sample Number: TC022136
Coal, SCCHG21099BC

Certificate Of Analysis

Ultimate Analyses

Dry Basis

As-Received

Carbon, %	72.69	69.87
Hydrogen, %	5.15	5.39
Nitrogen, %	1.54	1.49
Sulfur, %	0.59	0.56
Ash, %	9.71	9.34
Oxygen (by difference), %	10.32	13.36
Total Moisture, %		3.87

KJK/100

Respectfully Submitted
A. J. EDMOND COMPANY

Form 210f1

by


K. J. Kumke



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November 18, 1999

Air Products Stockton Cogen
1010 Zephyr Street
Stockton, CA 95206

Project Number: TC00557
Sample Number: TC022136
Coal #SCCHG21099BC

Attn: Ms. Tara Keefover

LABORATORY ANALYSES

As Received Basis:

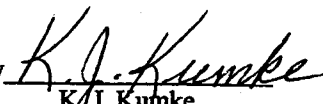
Moisture, %	3.87
-------------	------

Dry Basis:

Volatile Matter, %	39.51
Ash, %	9.71
Fixed Carbon, %	50.78
Sulfur, %	0.59
Oxygen (by difference), %	10.32
Calorific Value, Gross, BTU/Lb	12935
Chlorine, ppm	612
Mercury, ppm	0.026

Respectfully Submitted

A. J. EDMOND COMPANY

by 
K.J. Kumke



A.J. EDMOND CO.

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Air Products Stockton Cogen
1010 Zephyr Street
Stockton, CA 95206

November 17, 1999

Project Number: TC00557
Sample Number: TC022137
Coal, SCCHG31099BC

Certificate Of Analysis

Ultimate Analyses

Dry Basis

As-Received

Carbon, %	70.42	67.07
Hydrogen, %	5.23	5.51
Nitrogen, %	1.40	1.33
Sulfur, %	0.56	0.53
Ash, %	12.78	12.17
Oxygen (by difference), %	9.62	13.39
Total Moisture, %		4.76

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Respectfully Submitted
A. J. EDMOND COMPANY

by K. J. Kumke
K. J. Kumke

Form 2.10f1



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November 18, 1999

Air Products Stockton Cogen
1010 Zephyr Street
Stockton, CA 95206

Project Number: TC00557
Sample Number: TC022137
Coal #SCCHG31099BC

Attn: Ms. Tara Keefover

LABORATORY ANALYSES

As Received Basis:

Moisture, %	1.16
-------------	------

Dry Basis:

Volatile Matter, %	39.74
Ash, %	12.78
Fixed Carbon, %	47.48
Sulfur, %	0.56
Oxygen (by difference), %	9.62
Calorific Value, Gross, BTU/Lb	12254
Chlorine, ppm	470
Mercury, ppm	0.029

Respectfully Submitted

A. J. EDMOND COMPANY

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K. J. Kumke



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November 19, 1999

Air Products Stockton Cogen
1010 Zephyr Street
Stockton, CA 95206

Project Number: TC00557
Sample Number: TC022139
Fluid Coke #SCCHG11099FC

Certificate Of Analysis

Ultimate Analyses

Dry Basis

As-Received

Carbon, %	91.67	89.10
Hydrogen, %	2.10	2.04
Nitrogen, %	2.92	2.84
Sulfur, %	2.36	2.29
Ash, %	0.40	0.39
Oxygen (by difference), %	0.548	0.533
Total Moisture, %		2.80

KJK/100

Respectfully Submitted
A. J. EDMOND COMPANY

Form 2.10f1

by


K. J. Kumke



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November 19, 1999

Air Products Stockton Cogen
1010 Zephyr Street
Stockton, CA 95206

Project Number: TC00557
Sample Number: TC022139
Tosco Fluid Coke SCCHG11099FC

Attn: Ms. Tara Keefover

LABORATORY ANALYSES

As Received Basis:

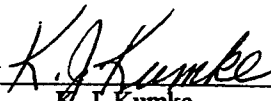
Moisture, %	2.80
-------------	------

Dry Basis:

Carbon, %	91.67
Oxygen (by difference), %	0.548
Calorific Value, Gross, BTU/Lb	14661
Chlorine, ppm	165
Mercury, ppm	0.045

Respectfully Submitted

A. J. EDMOND COMPANY

by 
K. J. Kumke



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November 19, 1999

Air Products Stockton Cogen
1010 Zephyr Street
Stockton, CA 95206

Project Number: TC00557
Sample Number: TC022140
Fluid Coke #SCCHG21099FC

Certificate Of Analysis

Ultimate Analyses

Carbon, %
Hydrogen, %
Nitrogen, %
Sulfur, %
Ash, %
Oxygen (by difference), %
Total Moisture, %

Dry Basis

92.11
2.14
2.22
2.14
0.42
0.120

As-Received

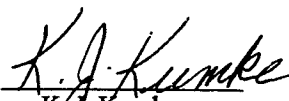
86.16
2.07
2.15
2.07
0.41
0.116
3.30

KJK/100

Respectfully Submitted
A. J. EDMOND COMPANY

Form 2.10f1

by


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November 19, 1999

Air Products Stockton Cogen
1010 Zephyr Street
Stockton, CA 95206

Project Number: TC00557
Sample Number: TC022140
Tosco Fluid Coke SCCHG21099FC

Attn: Ms. Tara Keefover

LABORATORY ANALYSES

As Received Basis:

Moisture, %	3.30
-------------	------

Dry Basis:

Carbon, %	92.11
Oxygen (by difference), %	0.120
Calorific Value, Gross, BTU/Lb	14710
Chlorine, ppm	142
Mercury, ppm	0.012

Respectfully Submitted

A. J. EDMOND COMPANY

by K. J. Kumke
K. J. Kumke



A.J. EDMOND CO.

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BARGE SURVEYS
SAMPLERS
SAMPLING SYSTEMS

November 19, 1999

Air Products Stockton Cogen
1010 Zephyr Street
Stockton, CA 95206

Project Number: TC00557
Sample Number: TC022141
Fluid Coke #SCCHG31099FC

Certificate Of Analysis

Ultimate Analyses

Carbon, %
Hydrogen, %
Nitrogen, %
Sulfur, %
Ash, %
Oxygen (by difference), %
Total Moisture, %

Dry Basis

91.45
2.26
2.76
2.40
0.39
0.744

As-Received

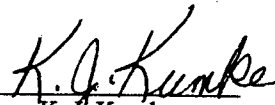
89.71
2.22
2.71
2.35
0.38
0.730
1.90

KJK/100

Respectfully Submitted
A. J. EDMOND COMPANY

Form 2.10f1

by


K. J. Kumke



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SAMPLERS
SAMPLING SYSTEMS

November 19, 1999

Air Products Stockton Cogen
1010 Zephyr Street
Stockton, CA 95206

Project Number: TC00557
Sample Number: TC022141
Tosco Fluid Coke SCCHG31099FC

Attn: Ms. Tara Keefover

LABORATORY ANALYSES

As Received Basis:

Moisture, %	1.90
-------------	------

Dry Basis:

Carbon, %	91.45
Oxygen (by difference), %	0.744
Calorific Value, Gross, BTU/Lb	14843
Chlorine, ppm	181
Mercury, ppm	0.031

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November 18, 1999

Air Products Stockton Cogen
1010 Zephyr Street
Stockton, CA 95206

Project Number: TC00557
Sample Number: TC022132
Limestone #SCCHG11099L

Attn: Ms. Tara Keefover

LABORATORY ANALYSES

As Received Basis:

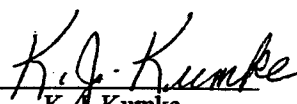
Dry Basis:

Calcium Oxide, CaO, % Oxide in ash	75.72
Magnesium Oxide, MgO, % Oxide in ash	5.91
Calcium Carbonate (CaCO ₃), %	81.22
Magnesium Carbonate (MgCO ₃), %	5.35
Mercury, ppm	0.0044

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November 18, 1999

Air Products Stockton Cogen
1010 Zephyr Street
Stockton, CA 95206

Project Number: TC00557
Sample Number: TC022133
Limestone #SCCHG21099L

Attn: Ms. Tara Keefover

LABORATORY ANALYSES

As Received Basis:

Dry Basis:

Calcium Oxide, CaO, % Oxide in ash	77.03
Magnesium Oxide, MgO, % Oxide in ash	3.32
Calcium Carbonate (CaCO ₃), %	82.46
Magnesium Carbonate (MgCO ₃), %	3.00
Mercury, ppm	0.0039

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November 18, 1999

Air Products Stockton Cogen
1010 Zephyr Street
Stockton, CA 95206

Project Number: TC00557
Sample Number: TC022134
Limestone #SCCHG31099L

Attn: Ms. Tara Keefover

LABORATORY ANALYSES

As Received Basis:

Dry Basis:

Calcium Oxide, CaO, % Oxide in ash	68.93
Magnesium Oxide, MgO, % Oxide in ash	4.71
Calcium Carbonate (CaCO ₃), %	75.04
Magnesium Carbonate (MgCO ₃), %	4.33
Mercury, ppm	0.0153

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November 18, 1999

Air Products Stockton Cogen
1010 Zephyr Street
Stockton, CA 95206

Project Number: TC00557
Sample Number: TC022142
Bottom Ash #SCCHG11099BA

Attn: Ms. Tara Keefover

LABORATORY ANALYSES

As Received Basis:

Dry Basis:

Carbon, %	3.35
CO ₂ , %	ND
Sulfur Trioxide, SO ₃ , %	18.64
Loss on Ignition, %	2.84
Calcium Oxide, CaO, % Oxide in ash	37.68
Magnesium Oxide, MgO, % Oxide in ash	1.97
Mercury, ppm	0.0045

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November 18, 1999

Air Products Stockton Cogen
1010 Zephyr Street
Stockton, CA 95206

Project Number: TC00557
Sample Number: TC022143
Bottom Ash #SCCHG21099BA

Attn: Ms. Tara Keefover

LABORATORY ANALYSES

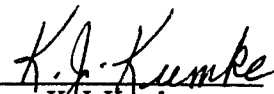
As Received Basis:

Dry Basis:

Carbon, %	5.77
CO ₂ , %	ND
Sulfur Trioxide, SO ₃ , %	12.17
Loss on Ignition, %	4.82
Calcium Oxide, CaO, % Oxide in ash	26.44
Magnesium Oxide, MgO, % Oxide in ash	1.54
Mercury, ppm	0.004

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November 18, 1999

Air Products Stockton Cogen
1010 Zephyr Street
Stockton, CA 95206

Project Number: TC00557
Sample Number: TC022144
Bottom Ash #SCCHG31099BA

Attn: Ms. Tara Keefover

LABORATORY ANALYSES

As Received Basis:

Dry Basis:

Carbon, %	1.36
CO ₂ , %	ND
Sulfur Trioxide, SO ₃ , %	18.43
Loss on Ignition, %	2.84
Calcium Oxide, CaO, % Oxide in ash	37.68
Magnesium Oxide, MgO, % Oxide in ash	1.97
Mercury, ppm	0.0045

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November 18, 1999

Air Products Stockton Cogen
1010 Zephyr Street
Stockton, CA 95206

Project Number: TC00557
Sample Number: TC022145
Fly Ash #SCCHG11099FA

Attn: Ms. Tara Keefover

LABORATORY ANALYSES

As Received Basis:

Dry Basis:

CO ₂ , %	ND
Sulfur Trioxide, SO ₃ , %	20.07
Loss on Ignition, %	25.59
Calcium Oxide, CaO, % Oxide in ash	27.59
Magnesium Oxide, MgO, % Oxide in ash	2.67
Mercury, ppm	0.019

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November 18, 1999

Air Products Stockton Cogen
1010 Zephyr Street
Stockton, CA 95206

Project Number: TC00557
Sample Number: TC022146
Fly Ash #SCCHG21099FA

Attn: Ms. Tara Keefover

LABORATORY ANALYSES


As Received Basis:

Dry Basis:

CO ₂ , %	ND
Sulfur Trioxide, SO ₃ , %	11.21
Loss on Ignition, %	22.47
Calcium Oxide, CaO, % Oxide in ash	35.17
Magnesium Oxide, MgO, % Oxide in ash	2.89
Mercury, ppm	0.057

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November 18, 1999

Air Products Stockton Cogen
1010 Zephyr Street
Stockton, CA 95206

Project Number: TC00557
Sample Number: TC022147
Fly Ash #SCCHG31099FA

Attn: Ms. Tara Keefover

LABORATORY ANALYSES


As Received Basis:

Dry Basis:

CO ₂ , %	ND
Sulfur Trioxide, SO ₃ , %	13.64
Loss on Ignition, %	14.08
Calcium Oxide, CaO, % Oxide in ash	33.71
Magnesium Oxide, MgO, % Oxide in ash	2.77
Mercury, ppm	0.064

Respectfully Submitted

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by 
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APPENDIX D

EMISSION CALCULATIONS

Appendix D.1

General Emissions Calculations

EMISSION CALCULATIONS

1. Sample Volume and Isokinetics

- a. Sample gas volume, dscf

$$V_{m\ std} = 0.0334 V_m \left(P_{bar} + \frac{H}{13.6} \right) \left(\frac{T_{ref}}{T_m} \right) (Y)$$

- b. Water vapor volume, scf

$$V_{w\ std} = 0.0472 V_{lc} \left(\frac{T_{ref}}{528\ ^\circ R} \right)$$

- c. Moisture content, nondimensional

$$B_{wo} = \frac{V_{w\ std}}{V_{m\ std} + V_{w\ std}}$$

- d. Stack gas molecular weight, lb/lb mole

$$MW_{dry} = 0.44 (\%CO_2) + 0.32 (\%O_2) + 0.28 (\%N_2)$$

$$MW_{wet} = MW_{dry} (1 - B_{wo}) + 18 (B_{wo})$$

- e. Absolute stack pressure, in Hg

$$P_s = P_{bar} + \frac{P_{sg}}{13.6}$$

- f. Stack velocity, ft/sec

$$V_s = 2.90 C_p \sqrt{\Delta PT_s} \sqrt{\left(\frac{29.92}{P_s} \right) \left(\frac{28.95}{MW_{wet}} \right)}$$

- g. Actual stack flow rate, wacfm

$$Q = (V_s)(A_s)(60)$$

- h. Standard stack gas flow rate, dscfm

$$Q_{sd} = Q (1 - B_{wo}) \left(\frac{T_{ref}}{T_s} \right) \left(\frac{P_s}{29.92} \right)$$

- i. Percent isokinetic

$$I = \left(\frac{17.32 (T_s) (V_{m\ std})}{(1 - B_{wo}) (\Theta) (V_s) (P_s) (D_n^2)} \right) \left(\frac{528\ ^\circ R}{T_{ref}} \right)$$

2. Particulate Emissions

- a. Grain loading, gr/dscf

$$C = 0.01543 \left(\frac{M_n}{V_{m \text{ std}}} \right)$$

- b. Grain loading at 12% CO
- ₂
- , gr/dscf

$$C_{12\% \text{ CO}_2} = C \left(\frac{12}{\% \text{ CO}_2} \right)$$

- c. Mass emissions, lb/hr

$$M = C(Q_{sd}) \frac{(60 \text{ min/hr})}{(7000 \text{ gr/lb})}$$

3. Gaseous Emissions, lb/hr

$$M = (ppm)(10^{-6}) \left(\frac{MW_i \text{ lb/lb mole}}{SV} \right) (Q_{sd})(60 \text{ min/hr})$$

where,

SV = specific molar volume of an ideal gas:

$$SV = 385.3 \text{ ft}^3/\text{lb mole} \quad \text{for } T_{ref} = 528 \text{ }^\circ R$$

$$SV = 379.5 \text{ ft}^3/\text{lb mole} \quad \text{for } T_{ref} = 520 \text{ }^\circ R$$

4. Emissions Rates, lb/10⁶ Btu

- a. Fuel factor at 68 °F, dscf/10
- ⁶
- Btu at 0% O
- ₂

$$F_{68} = \frac{10^6 [3.64(\%H) + 1.53(\%C) + 0.14(\%N) + 0.57(\%S) - 0.46(\%O_{2, \text{fuel}})]}{HHV, \text{ Btu/lb}}$$

- b. Fuel factor at 60 °F

$$F_{60} = F_{68} \left(\frac{520 \text{ }^\circ R}{528 \text{ }^\circ R} \right)$$

- c. Gaseous Emissions factor

$$\left(\frac{\text{lb}}{10^6 \text{ Btu}} \right)_i = (ppm)_i (10^{-6}) \left(\frac{MW_i \text{ lb}}{\text{lb mole}} \right) \left(\frac{1}{SV} \right) (F) \left(\frac{20.9}{20.9 - \%O_2} \right)$$

- d. Particulate emission factor

$$\left(\frac{\text{lb}}{10^6 \text{ Btu}} \right) = C \left(\frac{1 \text{ lb}}{7000 \text{ gr}} \right) (F) \left(\frac{20.9}{20.9 - \%O_2} \right)$$

Nomenclature:

A_s	=	stack area, ft ²
B_{wo}	=	flue gas moisture content
$C_{12\% CO_2}$	=	particulate grain loading, gr/dscf corrected to 12% CO ₂
C	=	particulate grain loading, gr/dscf
C_p	=	pitot calibration factor, dimensionless
D_n	=	nozzle diameter, in.
F	=	fuel F factor, dscf/10 ⁶ Btu at 0% O ₂
H	=	orifice pressure differential, iwg
I	=	% isokinetics
M_n	=	mass of collected particulate, mg
M_i	=	mass emissions of species i, lb/hr
MW	=	molecular weight of flue gas
MW_i	=	molecular weight of species i:
		NO _x : 46
		CO : 28
		SO _x : 64
		HC : 16
θ	=	sample time, min.
ΔP	=	average velocity head, iwg = $(\sqrt{\Delta P})^2$
P_{bar}	=	barometric pressure, in.Hg
P_s	=	stack absolute pressure, in.Hg
P_{sg}	=	stack static pressure, iwg

Nomenclature (Continued):

Q	= wet stack gas flow rate at actual conditions, wacfm
Q_{sd}	= dry stack gas flow rate at standard conditions, dscfm
SV	= specific molar volume of an ideal gas at standard conditions, ft ³ /lb mole
T_m	= meter temperature, °R
T_{ref}	= reference temperature, °R
T_s	= stack temperature, °R
V_s	= stack velocity, ft/sec
V_{lc}	= volume of liquid collected in impingers, ml
V_m	= dry meter volume uncorrected, dcf
$V_{m\ std}$	= dry meter volume at standard conditions, dscf
$V_{w\ std}$	= volume of water vapor at standard conditions, scf
Y	= meter calibration coefficient

TRACE SPECIES CALCULATIONS

- a. $\text{ng/sample train} = (\text{ng detected}) - (\text{ng in field blank})^*$
- b. $\text{ng/dscm} = \text{ng/sample train} \times (35.31/V_{\text{m std}})$
- c. $\text{ng/Nm}^3 \text{ at } 12\% \text{ CO}_2 = \text{ng/dscm} \times [T_{\text{ref}}(^{\circ}\text{R})/492^{\circ}\text{R}] \times (12\% \text{ CO}_2 / \% \text{ CO}_2)$
- d. $\text{lb/hr} = \text{ng/dscm} \times (1 \text{ g}/10^9 \text{ ng}) \times (1 \text{ lb}/454 \text{ g}) \times (1 \text{ m}^3/35.31 \text{ ft}^3) \times Q_{\text{sd}} \times (60 \text{ min/hr})$
(where Q_{sd} = standard gas flow rate, dscfm)

* This formula should be used with discretion. For example, very low blanks may merely indicate "noise," and might be disregarded. On the other hand, very high blank values may indicate sampling or analysis problems which should be investigated. It may not be acceptable to use a blank correction on some projects. For other projects a reagent blank may be more appropriate.

Appendix D.2

Spreadsheet Calculations

SOURCE TEST DATA SUMMARY

Client/Location.....	Air Products	*	Date.....	10/20/99
Test Number.....	1-Hg-Out	*	Data By.....	PG / EM
Test Method.....	Ontario-Hydro	*	Sample Location.....	Boiler Stack
Fuel.....	Coal / Coke	*	Unit.....	CFBC Boiler
Sample Train.....	N-3	*	Start/Stop Time.....	1330-1640

Pitot Factor	0.84	*	Reference Temp (F).....	60
Stack Area (sq ft).....	50.27	*	Meter Cal Factor.....	0.9960
Bar Press (in Hg).....	29.95	*	Sample Time (Min).....	160
Meter Vol (acf).....	84.978	*	Nozzle Diam (in).....	0.171
Stack Press (iwg).....	-0.81	*	Meter Temp (F).....	97.8
Vel Head (iwg).....	1.4818	*	Stack Temp (F).....	297.1
Liquid Vol (ml).....	102.7	*	Stack O ₂ (%).....	5.07
Meter Press (iwg).....	0.894	*	Stack CO ₂ (%).....	14.54

Std Sample Vol (SCF).....	79.102
Moisture Fraction.....	0.057
Stack Gas Mol Wt.....	29.82
Stack Gas Velocity (ft/sec).....	80.45
Stack Flow Rate (acfm).....	242620
Stack Flow Rate (wscfm).....	166438
Stack Flow Rate (dscfm).....	156965
Isokinetic Ratio (%).....	99.2

1-Hg-Out

Point No.	Stack Temp	Meter In	Meter Out	Delta P	Delta H	Meter Volume	Final	Liquid Volume Initial	Result
1	294	73	72	1.25	0.75	192.669			
	293	84	77	1.25	0.75		688.9	634.4	54.5
2	294	85	78	1.38	0.83		663.3	633.2	30.1
	293	92	80	1.38	0.83		623.0	619.7	3.3
3	293	94	81	1.55	0.93		623.7	621.6	2.1
	294	95	83	1.55	0.93		635.7	636.2	-0.5
4	294	97	84	1.51	0.91		687.6	687.1	0.5
	295	99	86	1.51	0.91		610.1	609.9	0.2
5	295	101	88	1.66	1.00		810.2	797.7	12.5
	295	101	91	1.66	1.00				
6	297	101	92	1.67	1.00		Average		102.7
	297	102	95	1.67	1.00				
7	297	104	99	1.52	0.91				
	297	104	101	1.52	0.91				
8	297	105	101	1.28	0.77				
	297	106	101	1.28	0.77	234.991			
Port Change									
1	297	104	103	1.01	0.61	235.089			
	297	105	102	1.01	0.61				
2	297	105	102	1.31	0.79				
	296	105	101	1.21	0.73				
3	297	106	101	1.38	0.83				
	299	107	101	1.38	0.83				
4	301	107	101	1.52	0.91				
	301	107	102	1.52	0.91				
5	301	106	101	1.69	1.01				
	301	104	100	1.64	0.98				
6	300	106	101	1.64	0.98				
	301	107	101	1.75	1.05				
7	299	107	101	1.76	1.06				
	299	107	100	1.76	1.06				
8	299	106	99	1.69	1.01				
	299	105	99	1.75	1.05	277.745			
Average	297.1 ✓	97.8 ✓		1.4818 RMS ✓	0.894 ✓	84.978 ✓		102.7 ✓	

Checked Rf Crosby
1-19-2000

SOURCE TEST DATA SUMMARY

Client/Location.....	Air Products	*	Date.....	10/21/99
Test Number.....	2-Hg-Out	*	Data By.....	PG / EM
Test Method.....	Ontario-Hydro	*	Sample Location.....	Boiler Stack
Fuel.....	Coal / Coke	*	Unit.....	CFBC Boiler
Sample Train.....	N-3	*	Start/Stop Time.....	1025-1325

Pitot Factor	0.84	*	Reference Temp (F).....	60
Stack Area (sq ft).....	50.27	*	Meter Cal Factor.....	0.9960
Bar Press (in Hg).....	29.90	*	Sample Time (Min).....	160
Meter Vol (acf).....	84.254	*	Nozzle Diam (in).....	0.171
Stack Press (iwg).....	-0.90	*	Meter Temp (F).....	87.7
Vel Head (iwg).....	1.472	*	Stack Temp (F).....	294.1
Liquid Vol (ml).....	100.4	*	Stack O ₂ (%).....	5.22
Meter Press (iwg).....	0.894	*	Stack CO ₂ (%).....	14.51

Std Sample Vol (SCF).....	79.741
Moisture Fraction.....	0.055
Stack Gas Mol Wt.....	29.84
Stack Gas Velocity (ft/sec).....	80.08
Stack Flow Rate (acfm).....	241503
Stack Flow Rate (wscfm).....	166016
Stack Flow Rate (dscfm).....	156837
Isokinetic Ratio (%).....	100.1

2-Hg-Out

Point No.	Stack Temp	Meter In	Meter Out	Delta P	Delta H	Meter Volume	Final	Liquid Volume Initial	Result
1	291	65	65	1.75	1.05	284.776			
	291	68	65	1.75	1.05		695.2	634.1	61.1
2	292	75	66	1.85	1.11		652.8	634.6	18.2
	293	80	67	1.88	1.13		623.6	620.4	3.2
3	295	81	68	1.88	1.13		623.9	622.0	1.9
	297	83	69	1.76	1.06		633.1	633.0	0.1
4	299	85	70	1.67	1.00		692.0	691.4	0.6
	299	87	72	1.67	1.00		611.1	611.9	-0.8
5	296	89	73	1.52	0.91		828.5	812.4	16.1
	295	89	74	1.52	0.91				
6	294	90	75	1.43	0.86		Average		100.4
	294	90	76	1.43	0.86				
7	293	92	77	1.15	0.69				
	292	90	79	1.15	0.69				
8	292	90	80	0.94	0.56				
	294	90	81	0.94	0.56	326.321			
Port Change									
1	293	86	85	1.25	0.75	326.421			
	294	92	86	1.30	0.78				
2	293	96	87	1.78	1.07				
	294	98	88	1.78	1.07				
3	294	99	91	1.01	0.61				
	294	98	92	1.01	0.61				
4	295	99	92	1.25	0.75				
	295	101	93	1.25	0.75				
5	294	102	93	1.75	1.05				
	295	104	94	1.81	1.09				
6	295	103	94	1.85	1.11				
	294	108	97	1.85	1.11				
7	293	106	97	1.51	0.91				
	293	110	98	1.51	0.91				
8	295	111	100	1.22	0.73				
	294	112	101	1.22	0.73	369.130			
Average	294.1 ✓	87.7 ✓		1.4723 ✓	0.894 ✓	84.254 ✓		100.4 ✓	

Rms

Checked 1-19-2000

Ry Guly

SOURCE TEST DATA SUMMARY

Client/Location.....	Air Products	*	Date.....	10/22/99
Test Number.....	3-Hg-Out	*	Data By.....	PG / EM
Test Method.....	Ontario-Hydro	*	Sample Location.....	Boiler Stack
Fuel.....	Coal / Coke	*	Unit.....	CFBC Boiler
Sample Train.....	N-3	*	Start/Stop Time.....	0850-1200

Pitot Factor	0.84	*	Reference Temp (F).....	60
Stack Area (sq ft).....	50.27	*	Meter Cal Factor.....	0.9960
Bar Press (in Hg).....	29.80	*	Sample Time (Min).....	160
Meter Vol (acf).....	86.161	*	Nozzle Diam (in).....	0.171
Stack Press (iwg).....	-0.70	*	Meter Temp (F).....	77.0
Vel Head (iwg).....	1.580	*	Stack Temp (F).....	292.5
Liquid Vol (ml).....	107.0	*	Stack O ₂ (%).....	5.15
Meter Press (iwg).....	0.955	*	Stack CO ₂ (%).....	14.50

Std Sample Vol (SCF).....	82.906
Moisture Fraction.....	0.057
Stack Gas Mol Wt.....	29.82
Stack Gas Velocity (ft/sec).....	83.02
Stack Flow Rate (acfm).....	250375
Stack Flow Rate (wscfm).....	171988
Stack Flow Rate (dscfm).....	162253
Isokinetic Ratio (%).....	100.6

3-Hg-Out

Point No.	Stack Temp	Meter In	Meter Out	Delta P	Delta H	Meter Volume	Final	Liquid Volume Initial	Result
1	290	56	56	1.35	0.81	372.442			
	289	65	57	1.35	0.81		705.1	651.6	53.5
2	290	70	57	1.57	0.94		646.0	613.8	32.2
	291	73	59	1.57	0.94		637.6	632.9	4.7
3	291	74	59	1.81	1.09		624.0	621.3	2.7
	291	75	61	1.81	1.09		632.0	632.7	-0.7
4	292	77	61	1.75	1.05		646.7	647.2	-0.5
	292	78	63	1.75	1.05		603.3	602.9	0.4
5	292	79	64	1.52	0.91		821.8	807.1	14.7
	292	79	65	1.52	0.91				
6	292	80	65	1.69	1.01		Average		107.0
	293	82	66	1.75	1.05				
7	292	83	68	1.61	0.97				
	292	83	69	1.57	0.94				
8	291	83	70	1.10	0.66				
	291	83	71	1.10	0.66	415.034			
Port Change									
1	292	73	73	1.88	1.13	415.920			
	294	80	72	1.88	1.13				
2	293	83	73	1.95	1.17				
	293	85	74	1.95	1.17				
3	293	90	77	1.90	1.14				
	293	91	78	1.90	1.14				
4	294	92	78	1.63	0.98				
	294	94	79	1.88	1.13				
5	294	93	80	1.67	1.00				
	294	94	81	1.67	1.00				
6	294	95	81	1.51	0.91				
	294	94	81	1.51	0.91				
7	294	93	82	1.29	0.77				
	295	95	83	1.29	0.77				
8	294	94	83	1.10	0.66				
	294	94	83	1.10	0.66	459.489			
Average	292.5 ✓		77.0 ✓	1.5802 ✓	0.955 ✓	86.161 ✓		107.0 ✓	

RMS

Checked 1-19-2000

KJ Cusky

SOURCE TEST DATA SUMMARY

Client/Location.....	Air Products	*	Date.....	10/20/99
Test Number.....	1-Hg-In	*	Data By.....	JP / EM
Test Method.....	Ontario-Hydro	*	Sample Location.....	Boiler Stack
Fuel.....	Coal / Coke	*	Unit.....	CFBC Boiler
Sample Train.....	N-3	*	Start/Stop Time.....	1324-1634

Pitot Factor	0.84	*	Reference Temp (F).....	60
Stack Area (sq ft).....	64.93	*	Meter Cal Factor.....	1.0180
Bar Press (in Hg).....	29.95	*	Sample Time (Min).....	168
Meter Vol (acf).....	57.221	*	Nozzle Diam (in).....	0.155
Stack Press (iwg).....	-12.40	*	Meter Temp (F).....	99.8
Vel Head (iwg).....	0.9811	*	Stack Temp (F).....	301.5
Liquid Vol (ml).....	73.3	*	Stack O ₂ (%).....	3.70
Meter Press (iwg).....	0.395	*	Stack CO ₂ (%).....	15.82

Std Sample Vol (SCF).....	54.180
Moisture Fraction.....	0.059
Stack Gas Mol Wt.....	29.93
Stack Gas Velocity (ft/sec).....	66.48
Stack Flow Rate (acfm).....	258994
Stack Flow Rate (wscfm).....	171608
Stack Flow Rate (dscfm).....	161455
Isokinetic Ratio (%).....	98.9

1-Hg-In

Point No.	Stack Temp	Meter In	Meter Out	Delta P	Delta H	Meter Volume	Final	Liquid Volume Initial	Result
A-63	300	94	94	0.83	0.33	682.422			
	301	95	94	0.85	0.34		625.8	584.5	41.3
A-62	301	96	95	0.87	0.35		641.7	629.0	12.7
	301	96	95	0.88	0.35		630.2	628.2	2.0
A-61	301	96	95	0.97	0.45		620.3	619.1	1.2
	300	96	95	0.93	0.37		627.8	629.4	-1.6
A-66	300	97	95	1.10	0.44		634.7	635.1	-0.4
	301	98	95	1.10	0.44		640.7	640.1	0.6
A-65	301	98	95	1.10	0.44		807.8	790.3	17.5
	301	99	95	1.10	0.44				
A-64	301	99	95	0.80	0.32		Average		73.3
	301	99	95	0.78	0.31	696.940			
Port Change									
B-6	304	95	95	1.00	0.40	698.743			
	305	99	95	1.10	0.44				
B-5	305	99	95	1.10	0.44				
	305	100	96	1.10	0.44				
B-4	303	100	96	0.99	0.40				
	303	100	96	0.97	0.39				
B-3	302	100	96	0.86	0.34				
	302	101	97	0.86	0.34				
B-2	302	101	97	0.84	0.34				
	301	102	98	0.85	0.34				
B-1	301	102	97	0.94	0.38				
	300	102	98	0.94	0.38	712.658			
Port Change									
C-6	301	103	98	1.10	0.44	713.415			
	301	103	98	1.10	0.44				
C-5	302	103	98	1.10	0.44				
	302	104	98	1.10	0.44				
C-4	302	104	98	0.93	0.37				
	302	104	99	0.93	0.37				
C-3	301	104	99	0.85	0.34				
	301	103	99	0.85	0.34				
C-2	301	104	99	0.90	0.36				
	301	104	100	0.88	0.35				
C-1	300	104	100	1.00	0.40				
	300	103	100	1.00	0.40	727.578			
Port Change									
D-6	300	105	100	1.30	0.52	729.518			
	300	106	101	1.20	0.48				
D-5	300	106	101	0.90	0.36				
	300	106	101	0.95	0.38				
D-4	300	106	101	1.00	0.40				
	303	106	102	1.10	0.44				
D-3	304	107	102	1.10	0.44				
	303	107	102	1.10	0.44				
D-2	304	107	103	1.00	0.40				
	302	107	103	1.00	0.40				
D-1	301	107	102	1.00	0.40				
	301	107	103	1.00	0.40	744.143			
Average	301.5 ✓	99.8 ✓		0.9811 ✓	0.395 ✓	57.221 ✓		73.3 ✓	

RMS

Checked 1-19-2000
KJ Gandy

SOURCE TEST DATA SUMMARY

Client/Location.....	Air Products	*	Date.....	10/21/99
Test Number.....	2-Hg-In	*	Data By.....	JP / EM
Test Method.....	Ontario-Hydro	*	Sample Location.....	Boiler Stack
Fuel.....	Coal / Coke	*	Unit.....	CFBC Boiler
Sample Train.....	N-3	*	Start/Stop Time.....	1012-1306

Pitot Factor	0.84	*	Reference Temp (F).....	60
Stack Area (sq ft).....	64.93	*	Meter Cal Factor.....	1.0180
Bar Press (in Hg).....	29.90	*	Sample Time (Min).....	168
Meter Vol (acf).....	57.706	*	Nozzle Diam (in).....	0.155
Stack Press (iwg).....	-12.00	*	Meter Temp (F).....	94.1
Vel Head (iwg).....	0.937	*	Stack Temp (F).....	298.2
Liquid Vol (ml).....	76.2	*	Stack O ₂ (%).....	4.36
Meter Press (iwg).....	0.376	*	Stack CO ₂ (%).....	14.99

Std Sample Vol (SCF).....	55.107
Moisture Fraction.....	0.060
Stack Gas Mol Wt.....	29.81
Stack Gas Velocity (ft/sec).....	64.97
Stack Flow Rate (acfm).....	253096
Stack Flow Rate (wscfm).....	168310
Stack Flow Rate (dscfm).....	158145
Isokinetic Ratio (%).....	102.7

2-Hg-In

Point No.	Stack Temp	Meter In	Meter Out	Delta P	Delta H	Meter Volume	Final	Liquid Volume Initial	Result
D-6	290	77	77	0.83	0.33	752.252			
	293	80	75	1.10	0.44		631.0	581.2	49.8
D-5	293	82	76	0.93	0.37		636.6	628.6	8.0
	293	84	77	0.90	0.36		627.1	627.2	-0.1
D-4	294	85	78	1.10	0.44		623.3	621.6	1.7
	295	85	78	1.10	0.44		630.7	630.4	0.3
D-3	296	87	80	1.10	0.44		632.4	632.5	-0.1
	298	88	81	1.10	0.44		643.1	644.1	-1.0
D-2	299	89	82	0.97	0.39		840.8	823.2	17.6
	299	90	82	0.97	0.39				
D-1	298	89	83	1.00	0.40				
	298	90	83	0.97	0.39	766.833			
Port Change									
C-6	306	92	85	1.00	0.40	767.825			
	303	92	85	1.00	0.40				
C-5	301	92	86	0.96	0.38				
	299	93	86	0.95	0.38				
C-4	297	93	87	0.85	0.34				
	297	94	87	0.85	0.34				
C-3	295	93	88	0.78	0.31				
	295	94	88	0.80	0.32				
C-2	296	94	88	0.83	0.33				
	296	95	89	0.87	0.35				
C-1	295	96	89	0.93	0.37				
	295	97	90	0.96	0.38	781.777			
Port Change									
B-6	300	98	91	1.00	0.40	782.638			
	302	100	92	1.00	0.40				
B-5	300	101	93	0.93	0.37				
	300	103	94	0.99	0.40				
B-4	300	104	95	0.94	0.38				
	299	104	95	0.95	0.38				
B-3	299	104	96	0.85	0.34				
	299	104	97	0.88	0.35				
B-2	298	104	98	0.82	0.33				
	299	104	98	0.85	0.34				
B-1	297	104	99	0.90	0.36				
	298	105	99	0.92	0.37	797.398			
Port Change									
A-6	301	106	101	1.10	0.44	798.383			
	302	107	100	1.10	0.44				
A-5	303	107	101	1.10	0.44				
	303	106	101	1.10	0.44				
A-4	302	106	101	0.75	0.30				
	301	105	102	0.75	0.30				
A-3	300	105	101	0.88	0.35				
	300	107	101	0.85	0.34				
A-2	299	107	103	0.85	0.34				
	299	107	103	0.85	0.34				
A-1	296	107	103	0.95	0.38				
	296	107	103	0.93	0.37	812.796			

Average 298.2 ✓ 94.1 ✓ 0.9367 ✓ 0.376 ✓ 57.706 ✓

RMS ✓

76.2 ✓

Checked 1-19-2000

K. C. C. C.

SOURCE TEST DATA SUMMARY

Client/Location.....	Air Products	*	Date.....	10/22/99
Test Number.....	3-Hg-In	*	Data By.....	JP / EM
Test Method.....	Ontario-Hydro	*	Sample Location.....	Boiler Stack
Fuel.....	Coal / Coke	*	Unit.....	CFBC Boiler
Sample Train.....	N-3	*	Start/Stop Time.....	0850-1149

Pitot Factor	0.84	*	Reference Temp (F).....	60
Stack Area (sq ft).....	64.93	*	Meter Cal Factor.....	1.0180
Bar Press (in Hg).....	29.80	*	Sample Time (Min).....	168
Meter Vol (acf).....	57.783	*	Nozzle Diam (in).....	0.155
Stack Press (iwg).....	-12.10	*	Meter Temp (F).....	81.7
Vel Head (iwg).....	0.988	*	Stack Temp (F).....	296.7
Liquid Vol (ml).....	76.9	*	Stack O ₂ (%).....	3.87
Meter Press (iwg).....	0.395	*	Stack CO ₂ (%).....	15.76

Std Sample Vol (SCF).....	56.257
Moisture Fraction.....	0.060
Stack Gas Mol Wt.....	29.92
Stack Gas Velocity (ft/sec).....	66.65
Stack Flow Rate (acfm).....	259646
Stack Flow Rate (wscfm).....	172369
Stack Flow Rate (dscfm).....	162071
Isokinetic Ratio (%).....	102.3

3-Hg-In

Point No.	Stack Temp	Meter In	Meter Out	Delta P	Delta H	Meter Volume		Liquid Volume	
							<i>Final</i>	<i>Initial</i>	<i>Result</i>
A-6	296	63	62	1.10	0.44	835.264			
	298	65	63	1.10	0.44		632.8	581.6	51.2
A-5	300	66	64	1.20	0.48		642.6	630.6	12.0
	300	68	65	1.20	0.48		628.5	627.7	0.8
A-4	300	70	66	0.80	0.32		618.5	617.7	0.8
	300	71	66	0.83	0.33		632.7	632.6	0.1
A-3	300	73	67	0.93	0.37		638.2	638.1	0.1
	299	74	68	0.95	0.38		642.6	642.7	-0.1
A-2	299	75	68	0.91	0.36		831.1	819.1	12.0
	299	76	69	0.90	0.36				
A-1	296	77	70	0.96	0.38		Average		76.9
	294	77	71	0.97	0.39	849.482			
Port Change									
B-6	300	79	72	1.10	0.44	850.028			
	301	80	73	1.10	0.44				
B-5	300	80	73	1.00	0.40				
	300	81	74	0.98	0.39				
B-4	299	81	75	0.96	0.38				
	299	82	75	0.96	0.38				
B-3	298	83	76	0.89	0.36				
	298	84	77	0.91	0.36				
B-2	298	84	78	0.85	0.34				
	296	85	79	0.85	0.34				
B-1	295	85	79	0.93	0.37				
	295	86	80	0.91	0.36	863.948			
Port Change									
C-6	295	87	81	1.10	0.44	864.828			
	296	87	81	1.10	0.44				
C-5	297	87	81	1.10	0.44				
	297	88	82	1.10	0.44				
C-4	297	89	83	0.81	0.32				
	297	89	83	0.90	0.36				
C-3	297	89	83	0.88	0.35				
	296	90	84	0.83	0.33				
C-2	295	90	84	0.88	0.35				
	296	90	85	0.88	0.35				
C-1	294	90	85	1.00	0.40				
	295	91	86	0.99	0.40	879.289			
Port-Change									
D-6	292	93	87	1.21	0.48	882.603			
	293	94	87	1.20	0.48				
D-5	293	94	88	1.00	0.40				
	294	94	88	0.98	0.39				
D-4	296	95	89	1.10	0.44				
	296	96	89	1.10	0.44				
D-3	296	96	90	1.10	0.44				
	296	96	91	1.10	0.44				
D-2	296	96	90	1.00	0.40				
	296	97	91	1.00	0.40				
D-1	291	98	92	0.95	0.38				
	290	98	92	0.95	0.38	897.787			
Average	296.7 ✓	81.7 ✓		0.9876 ✓	0.395 ✓	57.783 ✓		76.9 ✓	

RMS

Checked 1-19-2000
Rf Gwaly

MERCURY TEST RESULTS
AIR PRODUCTS / STOCKTON COGEN
CFBC BOILER OUTLET

Parameter	Test Runs				Sol'n Blank
	1-Hg-Out	2-Hg-Out	3-Hg-Out	AVERAGE	
Date	10/20/99	10/21/99	10/22/99		—
Flow Rate, dscfm	156,965	156,837	162,253	158,685	—
Sample Volume, dscf	79.10	79.74	82.91	80.58	—
O ₂ , % volume dry	5.07	5.22	5.15	5.15	—
CO ₂ , % volume dry	14.54	14.51	14.50	14.52	—
Mercury Lab					
Front half filter, ug	ND< 0.100	ND< 0.100	ND< 0.100	ND< 0.100	ND< 0.100
Front half rinse, ug	ND< 0.190	ND< 0.230	ND< 0.220	ND< 0.213	ND< 0.100
Impingers 1-3 contents, ug	ND< 0.190	ND< 0.220	ND< 0.210	ND< 0.207	ND< 0.020
Impinger 4 contents, ug	ND< 0.044	ND< 0.051	ND< 0.047	ND< 0.047	ND< 0.020
Impingers 5-7 contents, ug	ND< 0.150	ND< 0.180	ND< 0.190	ND< 0.173	ND< 0.020
Mercury, particle-bound					
Total ug/sample	ND< 0.290	ND< 0.330	ND< 0.320	ND< 0.313	ND< 0.200
*Blank corrected ug	ND< 0.090	ND< 0.130	ND< 0.120	ND< 0.113	ND< --
ug/dscm	ND< 0.129	ND< 0.146	ND< 0.136	ND< 0.137	ND< 0.088
* ug/dscm	ND< 0.040	ND< 0.058	ND< 0.051	ND< 0.050	ND< --
ug/dscm @ 3% O ₂	ND< 0.146	ND< 0.167	ND< 0.155	ND< 0.156	ND< 0.100
* ug/dscm @ 3% O ₂	ND< 0.045	ND< 0.066	ND< 0.058	ND< 0.056	ND< --
lb/hr	ND< 7.6E-05	ND< 8.6E-05	ND< 8.3E-05	ND< 8.2E-05	ND< 5.2E-05
* lb/hr	ND< 2.4E-05	ND< 3.4E-05	ND< 3.1E-05	ND< 2.9E-05	ND< --
Mercury, oxidized					
Total ug/sample	ND< 0.190	ND< 0.220	ND< 0.210	ND< 0.207	ND< 0.020
*Blank corrected ug	ND< 0.170	ND< 0.200	ND< 0.190	ND< 0.187	ND< --
ug/dscm	ND< 0.085	ND< 0.097	ND< 0.089	ND< 0.091	ND< 0.009
* ug/dscm	ND< 0.076	ND< 0.089	ND< 0.081	ND< 0.082	ND< --
ug/dscm @ 3% O ₂	ND< 0.096	ND< 0.111	ND< 0.102	ND< 0.103	ND< 0.010
* ug/dscm @ 3% O ₂	ND< 0.086	ND< 0.101	ND< 0.092	ND< 0.093	ND< --
lb/hr	ND< 5.0E-05	ND< 5.7E-05	ND< 5.4E-05	ND< 5.4E-05	ND< 5.2E-06
* lb/hr	ND< 4.5E-05	ND< 5.2E-05	ND< 4.9E-05	ND< 4.9E-05	ND< --
Mercury, elemental					
Total ug/sample	ND< 0.194	ND< 0.231	ND< 0.237	ND< 0.221	ND< 0.040
*Blank corrected ug	ND< 0.154	ND< 0.191	ND< 0.197	ND< 0.181	ND< --
ug/dscm	ND< 0.087	ND< 0.102	ND< 0.101	ND< 0.097	ND< 0.018
* ug/dscm	ND< 0.069	ND< 0.085	ND< 0.084	ND< 0.079	ND< --
ug/dscm @ 3% O ₂	ND< 0.098	ND< 0.117	ND< 0.115	ND< 0.110	ND< 0.020
* ug/dscm @ 3% O ₂	ND< 0.078	ND< 0.097	ND< 0.095	ND< 0.090	ND< --
lb/hr	ND< 5.1E-05	ND< 6.0E-05	ND< 6.1E-05	ND< 5.7E-05	ND< 1.0E-05
* lb/hr	ND< 4.0E-05	ND< 5.0E-05	ND< 5.1E-05	ND< 4.7E-05	ND< --
Mercury, Total					
Total ug/sample	ND< 0.674	ND< 0.781	ND< 0.767	ND< 0.741	ND< 0.260
*Blank corrected ug	ND< 0.414	ND< 0.521	ND< 0.507	ND< 0.481	ND< --
ug/dscm	ND< 0.301	ND< 0.346	ND< 0.327	ND< 0.324	ND< 0.114
* ug/dscm	ND< 0.185	ND< 0.231	ND< 0.216	ND< 0.210	ND< --
ug/dscm @ 3% O ₂	ND< 0.340	ND< 0.395	ND< 0.371	ND< 0.369	ND< 0.129
* ug/dscm @ 3% O ₂	ND< 0.209	ND< 0.263	ND< 0.245	ND< 0.239	ND< --
lb/hr	ND< 1.8E-04	ND< 2.0E-04	ND< 2.0E-04	ND< 1.9E-04	ND< 6.8E-05
* lb/hr	ND< 1.1E-04	ND< 1.4E-04	ND< 1.3E-04	ND< 1.3E-04	ND< --

* Results have been corrected for the solution blank analysis.

MERCURY TEST RESULTS
AIR PRODUCTS / STOCKTON COGEN
CFBC BOILER INLET

Parameter	Test Runs				Sol'n Blank
	1-Hg-In	2-Hg-In	3-Hg-In	AVERAGE	
Date	10/20/99	10/21/99	10/22/99		—
Flow Rate, dscfm	161,455	158,145	162,071	160,557	—
Sample Volume, dscf	54.180	55.107	56.257	55.18	—
O ₂ , % volume dry	3.70	4.36	3.87	3.98	—
CO ₂ , % volume dry	15.82	14.99	15.76	15.52	—
Mercury Lab					
Front half filter, ug	3.100	ND< 4.300	2.600	< 3.333	ND< 0.100
Front half rinse, ug	1.100	ND< 0.400	0.750	< 0.750	ND< 0.100
Impingers 1-3 contents, ug	ND< 0.210	ND< 0.220	ND< 0.210	ND< 0.213	ND< 0.020
Impinger 4 contents, ug	ND< 0.046	ND< 0.048	ND< 0.046	ND< 0.047	ND< 0.020
Impingers 5-7 contents, ug	ND< 0.160	ND< 0.170	ND< 0.180	ND< 0.170	ND< 0.020
Mercury, particle-bound					
Total ug/sample	4.200	ND< 4.700	3.350	< 4.083	ND< 0.200
*Blank corrected ug	4.000	ND< 4.500	3.150	< 3.883	-- --
ug/dscm	2.737	ND< 3.012	2.103	< 2.617	ND< 0.128
* ug/dscm	2.607	ND< 2.883	1.977	< 2.489	-- --
ug/dscm @ 3% O ₂	2.849	ND< 3.259	2.210	< 2.773	ND< 0.135
* ug/dscm @ 3% O ₂	2.713	ND< 3.121	2.078	< 2.637	-- --
lb/hr	1.7E-03	ND< 1.8E-03	1.3E-03	< 1.6E-03	ND< 7.7E-05
* lb/hr	1.6E-03	ND< 1.7E-03	1.2E-03	< 1.5E-03	-- --
Mercury, oxidized					
Total ug/sample	ND< 0.210	ND< 0.220	ND< 0.210	ND< 0.213	ND< 0.020
*Blank corrected ug	ND< 0.190	ND< 0.200	ND< 0.190	ND< 0.193	-- --
ug/dscm	ND< 0.137	ND< 0.141	ND< 0.132	ND< 0.137	ND< 0.013
* ug/dscm	ND< 0.124	ND< 0.128	ND< 0.119	ND< 0.124	-- --
ug/dscm @ 3% O ₂	ND< 0.142	ND< 0.153	ND< 0.139	ND< 0.145	ND< 0.014
* ug/dscm @ 3% O ₂	ND< 0.129	ND< 0.139	ND< 0.125	ND< 0.131	-- --
lb/hr	ND< 8.3E-05	ND< 8.3E-05	ND< 8.0E-05	ND< 8.2E-05	ND< 7.7E-06
* lb/hr	ND< 7.5E-05	ND< 7.6E-05	ND< 7.2E-05	ND< 7.4E-05	-- --
Mercury, elemental					
Total ug/sample	ND< 0.206	ND< 0.218	ND< 0.226	ND< 0.217	ND< 0.040
*Blank corrected ug	ND< 0.166	ND< 0.178	ND< 0.186	ND< 0.177	-- --
ug/dscm	ND< 0.134	ND< 0.140	ND< 0.142	ND< 0.139	ND< 0.026
* ug/dscm	ND< 0.108	ND< 0.114	ND< 0.117	ND< 0.113	-- --
ug/dscm @ 3% O ₂	ND< 0.140	ND< 0.151	ND< 0.149	ND< 0.147	ND< 0.027
* ug/dscm @ 3% O ₂	ND< 0.113	ND< 0.123	ND< 0.123	ND< 0.120	-- --
lb/hr	ND< 8.1E-05	ND< 8.3E-05	ND< 8.6E-05	ND< 8.3E-05	ND< 1.5E-05
* lb/hr	ND< 6.5E-05	ND< 6.8E-05	ND< 7.1E-05	ND< 6.8E-05	-- --
Mercury, Total					
Total ug/sample	< 4.616	ND< 5.138	< 3.786	< 4.513	ND< 0.260
*Blank corrected ug	< 4.356	ND< 4.878	< 3.526	< 4.253	-- --
ug/dscm	< 3.008	ND< 3.292	< 2.376	< 2.892	ND< 0.166
* ug/dscm	< 2.839	ND< 3.126	< 2.213	< 2.726	-- --
ug/dscm @ 3% O ₂	< 3.131	ND< 3.563	< 2.498	< 3.064	ND< 0.176
* ug/dscm @ 3% O ₂	< 2.954	ND< 3.383	< 2.326	< 2.888	-- --
lb/hr	< 1.8E-03	ND< 1.9E-03	< 1.4E-03	< 1.7E-03	ND< 1.0E-04
* lb/hr	< 1.7E-03	ND< 1.9E-03	< 1.3E-03	< 1.6E-03	-- --

* Results have been corrected for the solution blank analysis.

Note: Non-Detect for Run 2-Hg-In front half samples had a high reporting limit because there was so much particulate material in the samples.

Appendix D.3

Example Calculations



The Avogadro Group

SUBJECT Example Calculations - Mercury Speciation Project

JOB NO. 99057

Air Products / Stockton Cogen

SHEET NO. of

COMPUTED BY Kevin J. Crosby DATE 1-19-2000

CHECKED BY KJC

DATE 1-19-2000

Check Run# 1-Hg-In (V = verified spreadsheet)

$$a) V_{mstd} = 0.0334 V_m \left(P_{bar} + \frac{\Delta H}{13.6} \right) \left(\frac{T_{ref}}{T_m} \right) \gamma = 0.03342 * 57.221 * \left(29.95 + \frac{0.395}{13.6} \right) * \left(\frac{520}{559.8} \right) * 1.018$$

$$= \boxed{54.180} \checkmark$$

$$b) V_{wstd} = 0.0472 V_{gc} \left(\frac{T_{ref}}{528^{\circ}P} \right) = 0.0472 * 73.3 \text{ ml} * \left(\frac{520}{528} \right) = 3.4073 \text{ scf}$$

$$c) B_{wo} = \frac{V_{wstd}}{V_{mstd} + V_{wstd}} = \frac{3.4073}{54.180 + 3.4073} = \boxed{0.059} \checkmark$$

$$d) MW_{dry} = 0.44(\%CO_2) + 0.32(\%O_2) + 0.28(\%N_2) = 0.44(15.82) + 0.32(3.70) + 0.28(80.48)$$

$$= 30.68$$

$$MW_{wet} = MW_{dry} (1 - B_{wo}) + 18(B_{wo}) = 30.68 (1 - 0.059) + 18(0.059) = \boxed{29.93} \checkmark$$

$$e) P_s = P_{bar} + \frac{P_{sg}}{13.6} = 29.95 + \frac{-12.4}{13.6} = 29.038$$

$$f) V_s = 2.90 C_p \sqrt{\Delta P T_s} \sqrt{\left(\frac{29.92}{P_s} \right) \left(\frac{28.95}{MW_{wet}} \right)} = 2.90 * 0.84 * \sqrt{0.9811 * 761.5} * \sqrt{\left(\frac{29.92}{29.038} \right) \left(\frac{28.95}{29.93} \right)}$$

$$= \boxed{66.47} \checkmark (66.48)$$

$$g) Q = V_s A_s 60 = 66.48 * 64.93 \text{ ft}^2 * 60 = \boxed{258993} \checkmark (258994)$$

$$h) Q_{sd} = Q (1 - B_{wo}) \left(\frac{T_{ref}}{T_s} \right) \left(\frac{P_s}{29.92} \right) = 258993 (1 - 0.059) \left(\frac{520}{761.5} \right) \left(\frac{29.038}{29.92} \right) = \boxed{161516} \checkmark$$

(161455) -
(0.039% difference)

$$i) I = \left(\frac{17.32(T_s)(V_{mstd})}{(1 - B_{wo})(\theta)(V_s)(P_s)(D_u^2)} \right) \left(\frac{528}{T_{ref}} \right) = \left(\frac{17.32 * 761.5 * 54.180}{(1 - 0.059) 168 * 66.48 * 29.038 * (0.155)^2} \right) \left(\frac{528}{520} \right)$$

$$= \boxed{98.96\%} \checkmark (98.9)$$



The Avogadro Group

SUBJECT Example Calculations - Mercury Speciation Project JOB NO. 99057

Air Products / Stockton Cogen

SHEET NO. 01

COMPUTED BY Kevin J. Crosby DATE 1-19-2000 CHECKED BY KJC DATE 1-19-2000

Check Run # 1-Hg-In (✓ = verifies spreadsheet)

$$b) \mu\text{g}/\text{dscm} = \mu\text{g}/\text{sample} * (35.31 / V_{\text{mtd}})$$

$$\left\{ \begin{array}{l} \text{particle} \\ \text{-bound Hg} \end{array} \right\} = 4.20 * 35.31 / 54.180 = \boxed{2.737} \checkmark$$

$$\mu\text{g}/\text{dscm} @ 37.0_2 = \mu\text{g}/\text{dscm} * 179 / (20.9 - 9.0_2)$$

$$= 2.737 * 179 / (20.9 - 3.70) = \boxed{2.849} \checkmark$$

$$\text{lb}/\text{hr} = \mu\text{g}/\text{dscm} * \left(\frac{1\text{g}}{10^6 \mu\text{g}} \right) * \left(\frac{1\text{lb}}{454\text{g}} \right) * \left(\frac{1\text{m}^3}{35.31\text{ft}^3} \right) * Q_{\text{sd}} \frac{\text{ft}^3}{\text{min}} * 60 \frac{\text{min}}{\text{hr}}$$

$$= \frac{2.737 * 161455 * 60}{10^6 * 454 * 35.31} = \boxed{1.654 * 10^{-3}} \checkmark (1.7 * 10^{-3})$$

APPENDIX E

LABORATORY REPORTS

Appendix E.1

Mercury Analysis



Curtis & Tompkins, Ltd., Analytical Laboratories, Since 1878

2323 Fifth Street, Berkeley, CA 94710, Phone (510) 486-0900, Fax (510) 486-0532

A N A L Y T I C A L R E P O R T

Prepared for:

Avogadro Group
4085 Nelson Ave.
Suite E
Concord, CA 94520

Date: 03-DEC-99
Lab Job Number: 142489
Project ID: 99057
Location: Air Products, Stockton

Reviewed by: 

Reviewed by: 

This package may be reproduced only in its entirety.

Laboratory Number: **142489**
Client: **The Avogadro Group**
Location: **Air Products, Stockton**

Receipt Date: **10/25/99**

CASE NARRATIVE

This hardcopy data package contains sample and QC results for six trains, two field blanks, and three reagent blanks that were received on October 25, 1999. The samples were received intact and in good condition.

Analysis for mercury was performed using the Standard Test Method for Elemental, Oxidized, Particle-Bound, and Total Mercury in Flue Gas Generated from Coal-Fired Stationary Sources (Ontario Hydro Method), April 8, 1999. All samples were analyzed in duplicate. Please note that the detection limits (reporting limits) for the filter fractions vary depending on the total weight of the solid material on the filter.

No analytical problems were encountered.

CLIENT: Avogadro Group
PROJECT ID: 99057
LOCATION: Air Products, Stockton
MATRIX: Air

DATE REPORTED: 12/03/99

Metals Analytical Report

Mercury

Sample ID	Lab ID	Sample Date	Receive Date	Result (ug/Sample)	Reporting Limit (ug/Sample)	IDF	QC Batch	Method	Analysis Date
1-HG-I FILTER	142489-001	10/20/99	10/25/99	3.1	0.93	1	52056	EPA 7471	11/16/99
1-HG-I FRONT RINSE	142489-002	10/20/99	10/25/99	1.1	0.34	1	52057	EPA 7471	11/16/99
1-HG-I IMP 1-3	142489-003	10/20/99	10/25/99	ND	0.21	1	51975	EPA 7471	11/12/99
1-HG-I IMP 4	142489-004	10/20/99	10/25/99	ND	0.046	1	52003	EPA 7471	11/14/99
1-HG-I IMP 5-7	142489-005	10/20/99	10/25/99	ND	0.16	1	52004	EPA 7471	11/14/99
1-HG-O FILTER	142489-006	10/20/99	10/25/99	ND	0.10	1	52056	EPA 7471	11/16/99
1-HG-O FRONT RINSE	142489-007	10/20/99	10/25/99	ND	0.19	1	52057	EPA 7471	11/16/99
1-HG-O IMP 1-3	142489-008	10/20/99	10/25/99	ND	0.19	1	51975	EPA 7471	11/12/99
1-HG-O IMP 4	142489-009	10/20/99	10/25/99	ND	0.044	1	52003	EPA 7471	11/14/99
1-HG-O IMP 5-7	142489-010	10/20/99	10/25/99	ND	0.15	1	52004	EPA 7471	11/14/99
2-HG-I FILTER	142489-011	10/21/99	10/25/99	ND	4.3	1	52056	EPA 7471	11/16/99
2-HG-I FRONT RINSE	142489-012	10/21/99	10/25/99	ND	0.40	1	52057	EPA 7471	11/16/99
2-HG-I IMP 1-3	142489-013	10/21/99	10/25/99	ND	0.22	1	51975	EPA 7471	11/12/99
2-HG-I IMP 4	142489-014	10/21/99	10/25/99	ND	0.048	1	52003	EPA 7471	11/14/99
2-HG-I IMP 5-7	142489-015	10/21/99	10/25/99	ND	0.17	1	52004	EPA 7471	11/14/99
2-HG-O FILTER	142489-016	10/21/99	10/25/99	ND	0.10	1	52056	EPA 7471	11/16/99

ND = Not detected at or above reporting limit



CLIENT: Avogadro Group
 PROJECT ID: 99057
 LOCATION: Air Products, Stockton
 MATRIX: Air

DATE REPORTED: 12/03/99

Metals Analytical Report

Mercury

Sample ID	Lab ID	Sample Date	Receive Date	Result (ug/Sample)	Reporting Limit (ug/Sample)	IDF	QC Batch	Method	Analysis Date
2-HG-O FRONT RINSE	142489-017	10/21/99	10/25/99	ND	0.23	1	52057	EPA 7471	11/16/99
2-HG-O IMP 1-3	142489-018	10/21/99	10/25/99	ND	0.22	1	51975	EPA 7471	11/12/99
2-HG-O IMP 4	142489-019	10/21/99	10/25/99	ND	0.051	1	52003	EPA 7471	11/14/99
2-HG-O IMP 5-7	142489-020	10/21/99	10/25/99	ND	0.18	1	52004	EPA 7471	11/14/99
3-HG-I FILTER	142489-021	10/22/99	10/25/99	2.6	0.81	1	52056	EPA 7471	11/16/99
3-HG-I FRONT RINSE	142489-022	10/22/99	10/25/99	0.75	0.37	1	52057	EPA 7471	11/16/99
3-HG-I IMP 1-3	142489-023	10/22/99	10/25/99	ND	0.21	1	51975	EPA 7471	11/12/99
3-HG-I IMP 4	142489-024	10/22/99	10/25/99	ND	0.046	1	52003	EPA 7471	11/14/99
3-HG-I IMP 5-7	142489-025	10/22/99	10/25/99	ND	0.18	1	52004	EPA 7471	11/14/99
3-HG-O FILTER	142489-026	10/22/99	10/25/99	ND	0.10	1	52056	EPA 7471	11/16/99
3-HG-O FRONT RINSE	142489-027	10/22/99	10/25/99	ND	0.22	1	52057	EPA 7471	11/16/99
3-HG-O IMP 1-3	142489-028	10/22/99	10/25/99	ND	0.21	1	51975	EPA 7471	11/12/99
3-HG-O IMP 4	142489-029	10/22/99	10/25/99	ND	0.047	1	52003	EPA 7471	11/14/99
3-HG-O IMP 5-7	142489-030	10/22/99	10/25/99	ND	0.19	1	52004	EPA 7471	11/14/99
FB-HG-I FILTER	142489-031	10/21/99	10/25/99	ND	0.10	1	52056	EPA 7471	11/16/99
FB-HG-I FRONT RINSE	142489-032	10/21/99	10/25/99	ND	0.21	1	52057	EPA 7471	11/16/99

ND = Not detected at or above reporting limit



CLIENT: Avogadro Group
 PROJECT ID: 99057
 LOCATION: Air Products, Stockton
 MATRIX: Air

DATE REPORTED: 12/03/99

Metals Analytical Report

Mercury

Sample ID	Lab ID	Sample Date	Receive Date	Result (ug/Sample)	Reporting Limit (ug/Sample)	IDF	QC Batch	Method	Analysis Date
FB-HG-I IMP 1-3	142489-033	10/21/99	10/25/99	ND	0.13	1	51975	EPA 7471	11/12/99
FB-HG-I IMP 4	142489-034	10/21/99	10/25/99	ND	0.042	1	52003	EPA 7471	11/14/99
FB-HG-I IMP 5-7	142489-035	10/21/99	10/25/99	ND	0.17	1	52004	EPA 7471	11/14/99
FB-HG-0 FILTER	142489-036	10/21/99	10/25/99	ND	0.10	1	52056	EPA 7471	11/16/99
FB-HG-0 FRONT RINSE	142489-037	10/21/99	10/25/99	ND	0.16	1	52057	EPA 7471	11/16/99
FB-HG-0 IMP 1-3	142489-038	10/21/99	10/25/99	ND	0.15	1	51975	EPA 7471	11/12/99
FB-HG-0 IMP 4	142489-039	10/21/99	10/25/99	ND	0.046	1	52003	EPA 7471	11/14/99
FB-HG-0 IMP 5-7	142489-040	10/21/99	10/25/99	ND	0.19	1	52004	EPA 7471	11/14/99
RB 10/20 CONT 7	142489-041	10/20/99	10/25/99	ND	0.10	1	52057	EPA 7471	11/16/99
RB 10/20 CONT 8	142489-042	10/20/99	10/25/99	ND	0.020	1	51975	EPA 7471	11/12/99
RB 10/20 CONT 9	142489-043	10/20/99	10/25/99	ND	0.020	1	52003	EPA 7471	11/14/99
RB 10/20 CONT 10	142489-044	10/20/99	10/25/99	ND	0.020	1	52004	EPA 7471	11/14/99
RB 10/20 CONT 11	142489-045	10/20/99	10/25/99	ND	0.040	1	51975	EPA 7471	11/12/99
RB 10/20 CONT 12	142489-046	10/20/99	10/25/99	ND	0.10	1	52056	EPA 7471	11/16/99
RB 10/21 CONT 7	142489-047	10/20/99	10/25/99	ND	0.10	1	52057	EPA 7471	11/16/99
RB 10/21 CONT 8	142489-048	10/20/99	10/25/99	ND	0.020	1	51975	EPA 7471	11/12/99

ND = Not detected at or above reporting limit



CLIENT: Avogadro Group
 PROJECT ID: 99057
 LOCATION: Air Products, Stockton
 MATRIX: Air

DATE REPORTED: 12/03/99

Metals Analytical Report

Mercury

Sample ID	Lab ID	Sample Date	Receive Date	Result (ug/Sample)	Reporting Limit (ug/Sample)	IDF	QC Batch	Method	Analysis Date
RB 10/21 CONT 9	142489-049	10/20/99	10/25/99	ND	0.020	1	52003	EPA 7471	11/14/99
RB 10/21 CONT 10	142489-050	10/20/99	10/25/99	ND	0.020	1	52004	EPA 7471	11/14/99
RB 10/21 CONT 11	142489-051	10/20/99	10/25/99	ND	0.040	1	51975	EPA 7471	11/12/99
RB 10/21 CONT 12	142489-052	10/20/99	10/25/99	ND	0.10	1	52056	EPA 7471	11/16/99
RB 10/22 CONT 7	142489-053	10/22/99	10/25/99	ND	0.10	1	52057	EPA 7471	11/16/99
RB 10/22 CONT 8	142489-054	10/22/99	10/25/99	ND	0.020	1	51975	EPA 7471	11/12/99
RB 10/22 CONT 9	142489-055	10/22/99	10/25/99	ND	0.020	1	52003	EPA 7471	11/14/99
RB 10/22 CONT 10	142489-056	10/22/99	10/25/99	ND	0.020	1	52004	EPA 7471	11/14/99
RB 10/22 CONT 11	142489-057	10/22/99	10/25/99	ND	0.040	1	51975	EPA 7471	11/12/99
RB 10/22 CONT 12	142489-058	10/22/99	10/25/99	ND	0.10	1	52056	EPA 7471	11/16/99
ND = Not detected at or above reporting limit									





CLIENT: Avogadro Group
JOB NUMBER: 142489

DATE REPORTED: 12/03/99

BATCH QC REPORT
PREP BLANK

Compound	Result	Reporting Units		IDF	QC Batch	Method	Analysis Date
		Limit					
Mercury	ND	0.2	ug	1	51975	EPA 7471	11/12/99
Mercury	ND	0.02	ug	1	52003	EPA 7471	11/14/99
Mercury	ND	0.02	ug	1	52004	EPA 7471	11/14/99
Mercury	ND	0.1	ug	1	52056	EPA 7471	11/16/99
Mercury	ND	0.02	ug	1	52057	EPA 7471	11/16/99

ND = Not Detected at or above reporting limit



CLIENT: Avogadro Group
JOB NUMBER: 142489

DATE REPORTED: 12/03/99

BATCH QC REPORT
SAMPLE DUPLICATE

Compound	Sample	Sample Result	Duplicate Result	Units	RPD %	RPD Limit	QC Batch	Method	Analysis Date
Mercury	142489-038	<0.150	<0.150	ug	NC	20	51975	EPA 7471	11/12/99
Mercury	142489-033	<0.130	<0.130	ug	NC	20	51975	EPA 7471	11/12/99
Mercury	142489-028	<0.214	<0.214	ug	NC	20	51975	EPA 7471	11/12/99
Mercury	142489-008	<0.195	<0.195	ug	NC	20	51975	EPA 7471	11/12/99
Mercury	142489-018	<0.216	<0.216	ug	NC	20	51975	EPA 7471	11/12/99
Mercury	142489-023	<0.212	<0.212	ug	NC	20	51975	EPA 7471	11/12/99
Mercury	142489-013	<0.216	<0.216	ug	NC	20	51975	EPA 7471	11/12/99
Mercury	142489-003	<0.210	<0.210	ug	NC	20	51975	EPA 7471	11/12/99
Mercury	142489-003	<0.210	<0.210	ug	NC	20	51975	EPA 7471	11/12/99
Mercury	142489-024	<0.046	<0.046	ug	NC	20	52003	EPA 7471	11/14/99
Mercury	142489-034	<0.042	<0.042	ug	NC	20	52003	EPA 7471	11/14/99
Mercury	142489-039	<0.046	<0.046	ug	NC	20	52003	EPA 7471	11/14/99
Mercury	142489-029	<0.047	<0.047	ug	NC	20	52003	EPA 7471	11/14/99
Mercury	142489-019	<0.051	<0.051	ug	NC	20	52003	EPA 7471	11/14/99
Mercury	142489-014	<0.048	<0.048	ug	NC	20	52003	EPA 7471	11/14/99
Mercury	142489-004	<0.046	<0.046	ug	NC	20	52003	EPA 7471	11/14/99
Mercury	142489-009	<0.044	<0.044	ug	NC	20	52003	EPA 7471	11/14/99
Mercury	142489-035	<0.168	<0.168	ug	NC	20	52004	EPA 7471	11/14/99
Mercury	142489-040	<0.190	<0.190	ug	NC	20	52004	EPA 7471	11/14/99
Mercury	142489-025	<0.180	<0.180	ug	NC	20	52004	EPA 7471	11/14/99
Mercury	142489-020	<0.178	<0.178	ug	NC	20	52004	EPA 7471	11/14/99
Mercury	142489-030	<0.194	<0.194	ug	NC	20	52004	EPA 7471	11/14/99
Mercury	142489-010	<0.150	<0.150	ug	NC	20	52004	EPA 7471	11/14/99
Mercury	142489-015	<0.170	<0.170	ug	NC	20	52004	EPA 7471	11/14/99
Mercury	142489-005	<0.160	<0.160	ug	NC	20	52004	EPA 7471	11/14/99
Mercury	142489-005	<0.160	<0.160	ug	NC	20	52004	EPA 7471	11/14/99
Mercury	142489-006	<0.100	<0.100	ug	NC	20	52056	EPA 7471	11/16/99
Mercury	142489-006	<0.100	<0.100	ug	NC	20	52056	EPA 7471	11/16/99
Mercury	142489-052	<0.100	<0.100	ug	NC	20	52056	EPA 7471	11/16/99
Mercury	142489-058	<0.100	<0.100	ug	NC	20	52056	EPA 7471	11/16/99
Mercury	142489-036	<0.100	<0.100	ug	NC	20	52056	EPA 7471	11/16/99
Mercury	142489-016	<0.100	<0.100	ug	NC	20	52056	EPA 7471	11/16/99
Mercury	142489-046	<0.100	<0.100	ug	NC	20	52056	EPA 7471	11/16/99
Mercury	142489-031	<0.100	<0.100	ug	NC	20	52056	EPA 7471	11/16/99
Mercury	142489-026	<0.100	<0.100	ug	NC	20	52056	EPA 7471	11/16/99
Mercury	142489-021	2.614	2.553	ug	2	20	52056	EPA 7471	11/16/99
Mercury	142489-001	3.058	2.927	ug	4	20	52056	EPA 7471	11/16/99
Mercury	142489-011	<4.329	<4.329	ug	NC	20	52056	EPA 7471	11/16/99
Mercury	142489-047	<0.100	<0.100	ug	NC	20	52057	EPA 7471	11/16/99
Mercury	142489-053	<0.100	<0.100	ug	NC	20	52057	EPA 7471	11/16/99
Mercury	142489-041	<0.100	<0.100	ug	NC	20	52057	EPA 7471	11/16/99
Mercury	142489-037	<0.160	<0.160	ug	NC	20	52057	EPA 7471	11/16/99
Mercury	142489-007	<0.192	<0.192	ug	NC	20	52057	EPA 7471	11/16/99
Mercury	142489-032	<0.206	<0.206	ug	NC	20	52057	EPA 7471	11/16/99
Mercury	142489-017	<0.228	<0.228	ug	NC	20	52057	EPA 7471	11/16/99
Mercury	142489-017	<0.228	<0.228	ug	NC	20	52057	EPA 7471	11/16/99
Mercury	142489-027	<0.222	<0.222	ug	NC	20	52057	EPA 7471	11/16/99
Mercury	142489-012	<0.400	<0.400	ug	NC	20	52057	EPA 7471	11/16/99
Mercury	142489-022	0.754	0.7416	ug	2	20	52057	EPA 7471	11/16/99
Mercury	142489-002	1.143	0.9215	ug	21*	20	52057	EPA 7471	11/16/99

* = Out of Limits
NC = Not Calculable



CLIENT: Avogadro Group
JOB NUMBER: 142489

DATE REPORTED: 12/03/99

BATCH QC REPORT
BLANK SPIKE / BLANK SPIKE DUPLICATE

Compound	Spike Amount	BS Result	BSD Result	Units	BS% Rec.	BSD% Rec.	Rec. Limits	RPD %	RPD Limit	QC Batch	Method	Analysis Date
Mercury	5.000	5.123	4.945	ug	103	99	80-120	4	20	51975	EPA 7471	11/12/99
Mercury	0.5000	0.5056	0.5105	ug	101	102	80-120	1	20	52003	EPA 7471	11/14/99
Mercury	0.5000	0.5107	0.523	ug	102	105	80-120	2	20	52004	EPA 7471	11/14/99
Mercury	0.5000	0.4965	0.4922	ug	99	98	80-120	1	20	52056	EPA 7471	11/16/99
Mercury	0.5000	0.4853	0.4846	ug	97	97	80-120	0	20	52057	EPA 7471	11/16/99

Appendix E.2

Laboratory Sample Logs

LABORATORY SAMPLE LOG

THE AVOGADRO GROUP

CLIENT
PROJECT NO.

Air Products
99057

Sample Date	Sample No.	Container Number	Sample Fraction	Sample Media	Recovered By	C of C By	Shipped By	Shipment Carrier	Airbill Number	Analytical Laboratory
-------------	------------	------------------	-----------------	--------------	--------------	-----------	------------	------------------	----------------	-----------------------

10/20/99	RB-Hg	7	<Blank>	0.1N HNO ₃	Dan Duncan	Dan Duncan	Dan Duncan	Lebo	Pick up	Cutistampens
10/20/99	RB-Hg	8	<Blank>	1N KCl	Dan Duncan					
10/20/99	RB-Hg	9	<Blank>	HNO ₃ /H ₂ O	Dan Duncan					
10/20/99	RB-Hg	10	<Blank>	H ₂ SO ₄ /KNO ₃	Dan Duncan					
10/20/99	RB-Hg	11	<Blank>	Hydrochloric Sulfuric	Dan Duncan					
10/20/99	RB-Hg	12	<Blank>	Filter (Quartz)	Dan Duncan					
10/20/99	1-Hg-I	1	Filter	Filter (Quartz)	Dan Duncan					
10/20/99	1-Hg-I	2	Front Half Ring	0.1N HNO ₃	Dan Duncan					
10/20/99	1-Hg-I	3	Imp 1-3, fines	1N KCl + H ₂ O	Dan Duncan					
10/20/99	1-Hg-I	4	Imp 4, fines	HNO ₃ /H ₂ O	Dan Duncan					
10/20/99	1-Hg-I	5	Imp 5-7, fines	H ₂ SO ₄ /KNO ₃	Dan Duncan					
10/20/99	1-Hg-O	1	Filter	Filter (Quartz)	Dan Duncan					
10/20/99	1-Hg-O	2	Front Half Ring	0.1N HNO ₃	Dan Duncan					
10/20/99	1-Hg-O	3	Imp 1-3, fines	1N KCl + H ₂ O	Dan Duncan					
10/20/99	1-Hg-O	4	Imp 4, fines	HNO ₃ /H ₂ O	Dan Duncan					
10/20/99	1-Hg-O	5	Imp 5-7, fines	H ₂ SO ₄ /KNO ₃	Dan Duncan					
10/20/99	RB-Hg	7	<Blank>	0.1N HNO ₃	Dan Duncan					
10/20/99	RB-Hg	8	<Blank>	0.1N HNO ₃	Dan Duncan					
10/20/99	RB-Hg	9	<Blank>	0.1N HNO ₃	Dan Duncan					
10/20/99	RB-Hg	10	<Blank>	H ₂ SO ₄ /KNO ₃	Dan Duncan					

LABORATORY SAMPLE LOG

THE AVOGADRO GROUP

CLIENT
PROJECT NO.

Per Products
99057

Sample Date	Sample No.	Container Number	Sample Fraction	Sample Media	Recovered By	C of C By	Shipped By	Shipment Carrier	Airbill Number	Analytical Laboratory
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10/21/99	RB-Hg	12	<Blank>	Filter (Quartz)	Dan Duncan	Dan Duncan	Dan Duncan	Cabo	Pick up	Curtis Flombers
10/21/99	RB-Hg	11	<Blank>	Hydrofluoric Sludge	Dan Duncan					
10/21/99	2-Hg-I	1	Filter	Filter (Quartz)	Dan Duncan					
10/21/99	2-Hg-I	2	Front Half Tank	0.1N HNO ₃	Dan Duncan					
10/21/99	2-Hg-I	3	Imp 1-3, rinses	1N KCl + rinses	Dan Duncan					
10/21/99	2-Hg-I	4	Imp 4, rinses	HNO ₃ H ₂ O ₂	Dan Duncan					
10/21/99	2-Hg-I	5	Imp 5-7, rinses	H ₂ SO ₄ /HNO ₃ sludge	Dan Duncan					
10/21/99	2-Hg-0	1	Filter	Filter (Quartz)	Dan Duncan					
10/21/99	2-Hg-0	2	Front Half Tank	0.1N HNO ₃	Dan Duncan					
10/21/99	2-Hg-0	3	Imp 1-3, rinses	1N KCl + rinses	Dan Duncan					
10/21/99	2-Hg-0	4	Imp 4, rinses	HNO ₃ H ₂ O ₂	Dan Duncan					
10/21/99	2-Hg-0	5	Imp 5-7, rinses	H ₂ SO ₄ /HNO ₃ sludge	Dan Duncan					
10/21/99	FB-Hg-I	1	Filter	Filter (Quartz)	Ernst Winbolla					
10/21/99	FB-Hg-I	2	Front Half Tank	0.1N HNO ₃	Dan Duncan					
10/21/99	FB-Hg-I	3	Imp 1-3, rinses	1N KCl + rinses	Dan Duncan					
10/21/99	FB-Hg-I	4	Imp 4, rinses	HNO ₃ H ₂ O ₂	Dan Duncan					
10/21/99	FB-Hg-I	5	Imp 5-7, rinses	H ₂ SO ₄ /HNO ₃ sludge	Dan Duncan					
10/21/99	FB-Hg-0	1	Filter	Filter (Quartz)	Ernst Winbolla					
10/21/99	FB-Hg-0	2	Front Half Tank	0.1N HNO ₃	Dan Duncan					
10/21/99	FB-Hg-0	3	Imp 1-3, rinses	1N KCl + rinses	Dan Duncan					
10/21/99	FB-Hg-0	4	Imp 4, rinses	HNO ₃ H ₂ O ₂	Dan Duncan					
10/21/99	FB-Hg-0	5	Imp 5-7, rinses	H ₂ SO ₄ /HNO ₃ sludge	Dan Duncan					
10/21/99	FB-Hg-0	1	Filter	Filter (Quartz)	Ernst Winbolla					
10/21/99	FB-Hg-0	2	Front Half Tank	0.1N HNO ₃	Dan Duncan					
10/21/99	FB-Hg-0	3	Imp 1-3, rinses	1N KCl + rinses	Dan Duncan					

LABORATORY SAMPLE LOG

THE AVOGADRO GROUP

CLIENT
PROJECT NO.

Air Products
99057

Sample Date	Sample No.	Container Number	Sample Fraction	Sample Media	Recovered By	C of C By	Shipped By	Shipment Carrier	Airbill Number	Analytical Laboratory
-------------	------------	------------------	-----------------	--------------	--------------	-----------	------------	------------------	----------------	-----------------------

10/21/99	FB-Hg-0	4	Imp 4, rinse	HNO ₃ /H ₂ O ₂	Dan Duncan	Dan Duncan	Dan Duncan	Cals	Pick up	Curtis Dapbas
10/21/99	FB-Hg-0	5	Imp 5-7, rinses	H ₂ SO ₄ /KNO ₃ + rinses	Dan Duncan					
10/22/99	FB-Hg	7	<Blank>	0.124 HNO ₃	Dan Duncan					
10/22/99	FB-Hg	8	<Blank>	1N KCl	Dan Duncan					
10/22/99	FB-Hg	9	<Blank>	HNO ₃ /H ₂ O ₂	Dan Duncan					
10/22/99	FB-Hg	10	<Blank>	H ₂ SO ₄ /KNO ₃	Dan Duncan					
10/22/99	FB-Hg	11	<Blank>	Hydrofluoric Sulfuric	Dan Duncan					
10/22/99	FB-Hg	12	<Blank>	Filter (Quartz)	Dan Duncan					
10/22/99	3-Hg-I	1	Filter	Filter (Quartz)	Dan Duncan					
10/22/99	3-Hg-I	2	Front half rinses	0.124 HNO ₃	Dan Duncan					
10/22/99	3-Hg-I	3	Imp 1-3, rinses	1N KCl + rinses	Dan Duncan					
10/22/99	3-Hg-I	4	Imp 4, rinse	HNO ₃ /H ₂ O ₂	Dan Duncan					
10/22/99	3-Hg-I	5	Imp 5-7, rinses	H ₂ SO ₄ /KNO ₃ + rinses	Dan Duncan					
10/22/99	3-Hg-0	1	Filter	Filter (Quartz)	Dan Duncan					
10/22/99	3-Hg-0	2	Front half rinses	0.124 HNO ₃	Dan Duncan					
10/22/99	3-Hg-0	3	Imp 1-3, rinses	1N KCl + rinses	Dan Duncan					
10/22/99	3-Hg-0	4	Imp 4, rinse	HNO ₃ /H ₂ O ₂	Dan Duncan					
10/22/99	3-Hg-0	5	Imp 5-7, rinses	H ₂ SO ₄ /KNO ₃ + rinses	Dan Duncan					
10/22/99	3-Hg-0	1	Filter	Filter (Quartz)	Dan Duncan					
10/22/99	3-Hg-0	2	Front half rinses	0.124 HNO ₃	Dan Duncan					
10/22/99	3-Hg-0	3	Imp 1-3, rinses	1N KCl + rinses	Dan Duncan					
10/22/99	3-Hg-0	4	Imp 4, rinse	HNO ₃ /H ₂ O ₂	Dan Duncan					
10/22/99	3-Hg-0	5	Imp 5-7, rinses	H ₂ SO ₄ /KNO ₃ + rinses	Dan Duncan					

DD

Run #: 1-Hg-O

Impinger	Init. Train reagent volume, ml	Condensed moisture volume, ml	Post-test Total liquid volume, ml	Container	Post-test		Total sample volume, ml
					Total liquid volume, ml	Rinse volume, ml	
1	100.0	54.5	154.5	2	0.0	100.0	100.0
2	100.0	30.1	130.1	3	387.9	175.0	562.9
3	100.0	3.3	103.3	4	102.1	50.0	152.1
4	100.0	2.1	102.1	5	300.2	95.0	395.2
5	100.0	-0.5	99.5				
6	100.0	0.5	100.5				
7	100.0	0.2	100.2				

Run #: 2-Hg-O

Impinger	Init. Train reagent volume, ml	Condensed moisture volume, ml	Post-test Total liquid volume, ml	Container	Post-test		Total sample volume, ml
					Total liquid volume, ml	Rinse volume, ml	
1	100.0	61.1	161.1	2	0.0	100.0	100.0
2	100.0	18.2	118.2	3	382.5	175.0	557.5
3	100.0	3.2	103.2	4	101.9	50.0	151.9
4	100.0	1.9	101.9	5	299.9	95.0	394.9
5	100.0	0.1	100.1				
6	100.0	0.6	100.6				
7	100.0	-0.8	99.2				

Run #: 3-Hg-O

Impinger	Init. Train reagent volume, ml	Condensed moisture volume, ml	Post-test Total liquid volume, ml	Container	Post-test		Total sample volume, ml
					Total liquid volume, ml	Rinse volume, ml	
1	100.0	53.5	153.5	2	0.0	100.0	100.0
2	100.0	32.2	132.2	3	390.4	175.0	565.4
3	100.0	4.7	104.7	4	102.7	50.0	152.7
4	100.0	2.7	102.7	5	299.2	95.0	394.2
5	100.0	-0.7	99.3				
6	100.0	-0.5	99.5				
7	100.0	0.4	100.4				

Run #: FB-Hg-O

Impinger	Init. Train reagent volume, ml	Condensed moisture volume, ml	Post-test Total liquid volume, ml	Container	Post-test		Total sample volume, ml
					Total liquid volume, ml	Rinse volume, ml	
1	100.0	0.0	100.0	2	0.0	100.0	100.0
2	100.0	0.0	100.0	3	300.0	175.0	475.0
3	100.0	0.0	100.0	4	100.0	50.0	150.0
4	100.0	0.0	100.0	5	300.0	95.0	395.0
5	100.0	0.0	100.0				
6	100.0	0.0	100.0				
7	100.0	0.0	100.0				

10

Run #: 1-Hg-I

Impinger	Init. Train reagent volume, ml	Condensed moisture volume, ml	Post-test Total liquid volume, ml
1	100	41.3	141.3
2	100	12.7	112.7
3	100	2.0	102.0
4	100	1.2	101.2
5	100	-1.6	98.4
6	100	-0.4	99.6
7	100	0.6	100.6

Container	Post-test Total liquid volume, ml	Rinse volume, ml	Total sample volume, ml
2	0.0	100.0	100.0
3	356.0	225.0	581.0
4	101.2	50.0	151.2
5	298.6	95.0	393.6

Run #: 2-Hg-I

Impinger	Init. Train reagent volume, ml	Condensed moisture volume, ml	Post-test Total liquid volume, ml
1	100.0	49.8	149.8
2	100.0	8.0	108.0
3	100.0	-0.1	99.9
4	100.0	1.7	101.7
5	100.0	0.3	100.3
6	100.0	-0.1	99.9
7	100.0	-1.0	99.0

Container	Post-test Total liquid volume, ml	Rinse volume, ml	Total sample volume, ml
2	0.0	100.0	100.0
3	357.7	225.0	582.7
4	101.7	50.0	151.7
5	299.2	95.0	394.2

Run #: 3-Hg-I

Impinger	Init. Train reagent volume, ml	Condensed moisture volume, ml	Post-test Total liquid volume, ml
1	100.0	51.2	151.2
2	100.0	12.0	112.0
3	100.0	0.8	100.8
4	100.0	0.8	100.8
5	100.0	0.1	100.1
6	100.0	0.1	100.1
7	100.0	-0.1	99.9

Container	Post-test Total liquid volume, ml	Rinse volume, ml	Total sample volume, ml
2	0.0	100.0	100.0
3	364.0	225.0	589.0
4	100.8	50.0	150.8
5	300.1	95.0	395.1

Run #: FB-Hg-I

Impinger	Init. Train reagent volume, ml	Condensed moisture volume, ml	Post-test Total liquid volume, ml
1	100.0	0.0	100.0
2	100.0	0.0	100.0
3	100.0	0.0	100.0
4	100.0	0.0	100.0
5	100.0	0.0	100.0
6	100.0	0.0	100.0
7	100.0	0.0	100.0

Container	Post-test Total liquid volume, ml	Rinse volume, ml	Total sample volume, ml
2	0.0	100.0	100.0
3	300.0	225.0	525.0
4	100.0	50.0	150.0
5	300.0	95.0	395.0

Appendix E.3

Certificate of Lot Analyses

**J.T. Baker Ultrex® Brand****Actual Analysis****WATER**

Product No. 6906

Lot No. N19539

<u>TEST</u>	<u>RESULT</u>	<u>TEST</u>	<u>RESULT</u>
Trace Impurities in parts per million		Lead (Pb)	< 1
Arsenic (As)	< 0.00001	Lithium (Li)	< 1
Boron (B)	< 0.00004	Lutetium (Lu)	< 1
Chloride (Cl)	< 0.02	Magnesium (Mg)	< 2
Phosphate (PO ₄)	< 0.01	Manganese (Mn)	< 1
Selenium (Se)	< 0.0001	Mercury (Hg)	< 8
Silicon (Si)	< 0.00008	Molybdenum (Mo)	< 2
Sulfate (SO ₄)	0.1	Neodymium (Nd)	< 1
Substances Reducing Permanganate	Passes Test	Nickel (Ni)	< 1
Trace Impurities in parts per trillion (pg/g)		Niobium (Nb)	< 1
Aluminum (Al)	12	Palladium (Pd)	< 1
Antimony (Sb)	< 1	Platinum (K)	< 1
Barium (Ba)	< 1	Potassium (K)	< 20
Beryllium (Be)	< 2	Praseodymium (Pr)	< 1
Bismuth (Bi)	< 2	Rhodium (Rh)	< 1
Cadmium (Cd)	< 2	Rubidium (Rb)	< 1
Calcium (Ca)	8	Ruthenium (Ru)	< 2
Cerium (Ce)	< 2	Samarium (Sm)	< 2
Cesium (Cs)	< 1	Scandium (Sc)	< 1
Chromium (Cr)	< 2	Silver (Ag)	< 1
Cobalt (Co)	< 1	Sodium (Na)	< 20
Copper (Cu)	< 2	Strontium (Sr)	< 1
Dysprosium (Dy)	< 2	Tantalum (Ta)	< 1
Erbium (Er)	< 1	Terbium (Tb)	< 1
Europium (Eu)	< 1	Thallium (Tl)	< 1
Gadolinium (Gd)	< 1	Thorium (Th)	< 1
Gallium (Ga)	< 1	Thulium (Tm)	< 1
Germanium (Ge)	< 1	Tin (Sn)	< 3
Gold (Au)	< 1	Titanium (Ti)	< 1
Holmium (Ho)	< 1	Tungsten (W)	< 1
Indium (In)	< 1	Uranium (U)	< 1
Iridium (Ir)	< 1	Vanadium (V)	< 1
Iron (Fe)	9	Ytterbium (Yb)	< 2
Lanthanum (La)	< 1	Zinc (Zn)	< 2
		Zirconium (Zr)	< 1

Signed

Director of Quality

J.T. BAKER

A Division of Mallinckrodt Baker, Inc.

Phillipsburg, NJ 08865 USA

Ph (908) 859-2151



J.T. Baker Ultrex® Brand

Actual Analysis

EM KCl soln on 10/31/99
CFR, R2-6

WATER

Product No. 6906

Lot No. M37540

<u>TEST</u>	<u>RESULT</u>	<u>TEST</u>	<u>RESULT</u>
Non-Metallic Impurities in parts per million ($\mu\text{g/g}$)		Lead (Pb)	22
Arsenic (As)	< 0.000001	Lithium (Li)	< 1
Boron (B)	< 0.00004	Lutetium (Lu)	2
Chloride (Cl)	< 0.01	Magnesium (Mg)	5
Phosphate (PO_4)	< 0.01	Manganese (Mn)	< 1
Selenium (Se)	< 0.001	Mercury (Hg)	104
Silicon (Si)	< 0.00008	Molybdenum (Mo)	< 1
Sulfate (SO_4)	< 0.05	Neodymium (Nd)	2
Substances Reducing Permanganate	Passes Test	Nickel (Ni)	3
Metallic Impurities in parts per trillion (pg/g)		Niobium (Nb)	2
Aluminum (Al)	< 2	Palladium (Pd)	2
Antimony (Sb)	3	Platinum (K)	< 1
Barium (Ba)	6	Potassium (K)	< 20
Beryllium (Be)	< 1	Praseodymium (Pr)	2
Bismuth (Bi)	8	Rhodium (Rh)	< 1
Cadmium (Cd)	< 1	Rubidium (Rb)	< 1
Calcium (Ca)	< 10	Ruthenium (Ru)	< 1
Cerium (Ce)	2	Samarium (Sm)	2
Cesium (Cs)	< 1	Scandium (Sc)	2
Chromium (Cr)	4	Silver (Ag)	< 1
Cobalt (Co)	< 1	Sodium (Na)	360
Copper (Cu)	1	Strontium (Sr)	< 1
Dysprosium (Dy)	2	Tantalum (Ta)	1
Erbium (Er)	2	Terbium (Tb)	2
Europium (Eu)	2	Thallium (Tl)	< 1
Gadolinium (Gd)	2	Thorium (Th)	< 1
Gallium (Ga)	< 1	Thulium (Tm)	2
Germanium (Ge)	< 1	Tin (Sn)	3
Gold (Au)	< 1	Titanium (Ti)	2
Holmium (Ho)	2	Tungsten (W)	2
Indium (In)	1	Uranium (U)	< 1
Iridium (Ir)	< 1	Vanadium (V)	< 1
Iron (Fe)	< 10	Ytterbium (Yb)	2
Lanthanum (La)	2	Zinc (Zn)	6
		Zirconium (Zr)	2

Signed

Kenn Weber

Director of Quality

J.T. BAKER

A Division of Mallinckrodt Baker, Inc.

Phillipsburg, NJ 08865 USA

Ph (908) 859-2151

GENERAL STORAGE

GENERAL STORAGE

GENERAL STORAGE

CAUTION! Causes Eye Irritation.
MAY CAUSE SKIN IRRITATION.
MAY BE HARMFUL IF SWALLOWED.
Keep container closed.
Do not breathe dust. Do not get
in eyes. Do not take internally.
Intended for laboratory and manufacturing use only. Not for drug,
food, or household use. For additional information, see MATERIAL
SAFETY DATA SHEET (MSDS)
for this material.



Made in Germany

L196

4938-1 50 G.
Potassium Chloride

Suprapur®

KCl FW 74.56

CAS 7447-40-7



55063

Certificate of Guarantee

Assay (an. titrimetric)	Co	max. 0.005 ppm
min. 99.5%	Cu	max. 0.005 ppm
Phosphate max. 5 ppm	Fe	max. 0.01 ppm
Sulfate max. 10 ppm	Pb	max. 0.01 ppm
Total Nitrogen max. 10 ppm	Li	max. 0.4 ppm
Al max. 0.01 ppm	Mg	max. 0.05 ppm
Ba max. 1 ppm	Mn	max. 0.01 ppm
Cd max. 0.005 ppm	Ni	max. 0.005 ppm
Cr max. 0.01 ppm	Na	max. 5 ppm
Ca max. 0.1 ppm	Sr	max. 0.1 ppm
	Ti	max. 0.01 ppm
	Zn	max. 0.005 ppm

EM SCIENCE

A Division of EM Industries, Inc.
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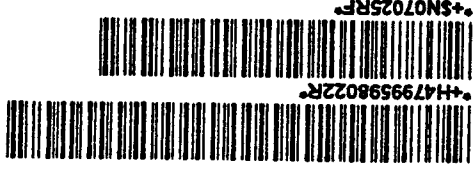
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POISON! DANGER!
 STRONG OXIDIZER. CONTACT WITH OTHER MATERIAL MAY CAUSE FIRE. CORROSIVE. LIQUID AND MIST MAY CAUSE SEVERE BURNS TO ALL BODY TISSUE. MAY BE FATAL IF SWALLOWED OR INHALED. INHALATION MAY CAUSE LUNG AND TOOTH DAMAGE.
 Do not get in eyes, on skin, or on clothing. Do not breathe vapors. Use only with adequate ventilation. Wash thoroughly after handling. Do not store near combustible materials. Store in a tightly closed container. Remove and wash contaminated clothing promptly. First Aid: In case of contact with eyes, flush with plenty of water for at least 15 minutes. If swallowed, do not induce vomiting. If inhaled, move to fresh air. If on skin, wash with plenty of water. Never give anything by mouth to an unconscious person. If breathing is difficult, give oxygen. In all cases get medical attention immediately.
 SEE MATERIAL SAFETY DATA SHEET

TOXIQUE! DANGER!
 OXYDANT FORT. EN CONTACT AVEC D'AUTRES MATIERES, IL PEUT PROVOQUER UN INCENDIE. CORROSIF. LE LIQUIDE ET LA BRUME PEUVENT CAUSER DE GRAVES BRULURES A TOUTES LES PARTIES DU CORPS. PEUT ETRE FATAL EN CAS D'INHALATION. L'INHALATION PEUT CAUSER DE GRAVES LESIONS AUX POUMONS ET A LA DENTURE.
 Ne pas se faire entrer dans les yeux, sur la peau, ou sur les vêtements. Ne pas respirer les vapeurs. Utiliser avec une ventilation adéquate. Se laver soigneusement après la manipulation. Ne pas stocker à proximité des matières combustibles. Stocker dans un récipient hermétiquement fermé. En cas de contact avec les yeux, rincer abondamment avec de l'eau pendant au moins 15 minutes. En cas d'ingestion, ne pas provoquer le vomissement. En cas d'inhalation, se déplacer dans un endroit où l'air est frais. En cas de contact avec la peau, se laver avec de l'eau. Ne jamais donner à boire à une personne inconsciente. En cas de difficulté à respirer, administrer de l'oxygène. Dans tous les cas, consulter un médecin immédiatement.



500 mL 9598-02
 Nitric Acid, 69.0-70.0%
 Acide Nitrique
 "BAKER NITRA-ANALYZED" Reagent
 For Trace Metal Analysis
 HNO₃ FW 63.01
 ACTUAL ANALYSIS, LOT
 N07025

Exceeds A.C.S. Specifications
 Assay (HNO₃) 69.0%
 Appearance Clear, colorless liquid
 Boiling Point (°C) 83
 Specific Gravity at 20°C 1.42

Trace Impurities (in ppm)	Limit (ppm)
Aluminum (Al)	< 0.5
Ammonium (NH ₄)	< 0.5
Barium (Ba)	< 0.5
Bismuth (Bi)	< 0.5
Boron (B)	< 0.5
Calcium (Ca)	< 0.5
Chromium (Cr)	< 0.5
Copper (Cu)	< 0.5
Iron (Fe)	< 0.5
Lead (Pb)	< 0.5
Magnesium (Mg)	< 0.5
Manganese (Mn)	< 0.5
Mercury (Hg)	< 0.5
Nickel (Ni)	< 0.5
Potassium (K)	< 0.5
Silver (Ag)	< 0.5
Sodium (Na)	< 0.5
Titanium (Ti)	< 0.5
Zinc (Zn)	< 0.5

Nitric Acid..... 69.0% 70.0%

BAKER SAF-T-DATA™ System

HEALTH HAZARD REACTIVITY CONTACT
 SEVERE 3 NONE 0 SEVERE 3 EXTREME 4

LABORATORY PROTECTIVE EQUIPMENT
 GOGGLES & SHIELD LAS COAT LAB COAT VENT HOOD PROPER GLOVES

STORAGE COLOR YELLOW

DOT Name: NITRIC ACID (WITH NOT MORE THAN 70% NITRIC ACID) UN2031
 CAS NO: 7697-37-2
 J. T. Baker NEUTRASORB® or TEA® Low Nitric acid neutralizers are recommended for spills of this product.
 MADE IN USA

J. T. Baker
 A Division of Mallinckrodt Baker, Inc.
 Phillipsburg, NJ 08865 USA
 PH (608) 859-2151

888884
 911610229
 R12

PRODUCTION NUMBER: 307260

Item Number: 321-1000

Item Description: JAR TALL CLEAR WM

Groups 1 and 2 are applicable

This is your Certificate of Analysis for I-CHEM Certified[™] product which has been prepared in accordance with I-CHEM Performance-Based Specifications. This product meets or exceeds analyte specifications established in the U.S. EPA "Specification and Guidance for Contaminant-free Sample Containers" for use in Superfund and other hazardous waste programs.

Group 1. Glass and HDPE Sample containers for use in the analysis of Metals					
Analyte	Detection Limit (µg/L)	Analyte	Detection Limit (µg/L)	Analyte	Detection Limit (µg/L)
Aluminum	<80	Calcium (all HDPE)	<100	Magnesium	<100
Antimony	<5	Chromium	<10	Manganese	<10
Arsenic	<2	Cobalt	<10	Mercury	<0.2
Barium	<20	Copper	<10	Nickel	<20
Beryllium	<0.5	Iron	<50	Potassium	<750
Cadmium	<1	Lead	<2	Potassium (all HDPE)	<100
Calcium	<500			Selenium	<2
				Silver	<5
				Sodium	<5000
				Sodium (all HDPE)	<100
				Thallium	<5
				Vanadium	<10
				Zinc	<10

In addition to the above analytes, NALGENE [®] containers are certified for these analytes:					
Analyte	Detection Limit (µg/L)	Analyte	Detection Limit (µg/L)	Analyte	Detection Limit (µg/L)
Chloride	<100	Fluoride	<20	Nitrite	<50
Cyanide	<10	Nitrate	<20	Paraquat (amber only)	<0.4
Disput (amber only)	<10			Sulfate	<100
				Sulfide	<30
				Sulfite	<1000

Group 2. Glass Sample Containers for use in the analysis of Semivolatiles and Pesticides/PCBs					
Compound	Quantitation Limit (µg/L)	Compound	Quantitation Limit (µg/L)	Compound	Quantitation Limit (µg/L)
Acenaphthene	<5	Acenaphthylene	<5	Anthracene	<5
Benzo(a)anthracene	<5	Benzo(a)pyrene	<5	Benzo(b)fluoranthene	<5
Benzo(k)fluoranthene	<5	Benzo(g,h,i)perylene	<5	Benzoic Acid	<20
Benzo(l)Acetol	<5	4-Bromophenylphenylether	<5	Butylbenzylphthalate	<5
4-Chloroaniline	<5	4-Chloro-3-methylphenol	<5	bis(2-Chloroethoxy)methane	<5
bis(2-Chloroethyl)ether	<5	4-Chlorophenylphenylether	<5	2-Chloronaphthalene	<5
2-Chlorophenol	<5	Di-n-octylphthalate	<5	Chrysene	<5
Di-n-butylphthalate	<5	1,2-Dichlorobenzene	<5	Dibenz(a,h)anthracene	<5
Dibenzofuran	<5	3,3'-Dichlorobenzidine	<5	1,4-Dichlorobenzene	<5
1,3-Dichlorobenzene	<5	Dimethylphthalate	<5	2,4-Dichlorophenol	<5
Diethylphthalate	<5	2,4-Dinitrophenol	<20	2,4-Dinitrotoluene	<5
4,6-Dinitro-2-methylphenol	<20	bis(2-Ethylhexyl)phthalate	<5	Fluoranthene	<5
2,6-Dinitrotoluene	<5	Hexachlorobenzene	<5	Hexachlorobutadiene	<5
Fluorene	<5	Hexachloromethane	<5	Indeno(1,2,3-cd)pyrene	<5
Hexachlorocyclopentadiene	<5	2-Methylnaphthalene	<5	2-Methylphenol	<5
Isophorone	<5	N-Nitrosodipropylamine	<5	3-Nitroaniline	<20
4-Methylphenol	<5	Naphthalene	<5	N-Nitrosodimethylamine	<5
4-Nitroaniline	<20	4-Nitrophenol	<20	Nitrobenzene	<5
N-Nitrosodiphenylamine	<5	Phenol	<5	Pentachlorophenol	<20
2-Nitrophenol	<5	2,4,5-Trichlorophenol	<20	Pyrene	<5
Phenanthrene	<5	Carbazole	<5	2,4,6-Trichlorophenol	<5
1,2,4-Trichlorobenzene	<5	Endosulfan II	<0.02	Aldrin	<0.01
Azobenzene	<5	Endosulfan Sulfate	<0.02	Alpha-BHC	<0.01
4,4-DDD	<0.02	Endrin	<0.02	Beta-BHC	<0.01
4,4-DDE	<0.02	Endrin Aldehyde	<0.02	Delta-BHC	<0.01
4,4-DDT	<0.02	Heptachlor	<0.01	Gamma-BHC	<0.01
Dieldrin	<0.02	Endrin Ketone	<0.02	Heptachlor Epoxide	<0.01
Endosulfan I	<0.01	Toxaphene	<0.30	Alpha-Chlordane	<0.01
Methoxychlor	<0.10	Arclor-1221	<0.20	Arclor-1016	<0.20
Gamma-Chlordane	<0.01	Arclor-1232	<0.20	Arclor-1242	<0.20
Arclor-1221	<0.20	Arclor-1254	<0.20	Arclor-1260	<0.20
Arclor-1248	<0.20	Arclor-1268	<0.20		
Arclor-1262	<0.20				

Group 3. Glass Sample Containers for use in the analysis of Volatiles					
Compound	Quantitation Limit (µg/L)	Compound	Quantitation Limit (µg/L)	Compound	Quantitation Limit (µg/L)
Acetone	<5	1,3-Dichloropropane	<1	Benzene	<1
2,2-Dichloropropane	<1	Bromobenzene	<1	1,2-Dichloropropane	<1
Permethyldichloromethane	<1	trans-1,3-Dichloropropane	<1	Bromoforn	<1
cis-1,3,4-Dichloropropene	<1	Bromomethane	<1	1,1-Dichloropropene	<1
2-Butanone	<5	Ethylbenzene	<1	tert-Butylbenzene	<1
Hexachlorobutadiene	<1	sec-Butylbenzene	<1	2-Hexanone	<5
n-Butylbenzene	<1	Isopropylbenzene	<1	Carbon Disulfide	<1
p-Isopropyltoluene	<1	Carbon Tetrachloride	<1	4-Methyl-2-pentanone	<5
Chlorobenzene	<1	Methylene Chloride	<2	Chloroethane	<1
Naphthalene	<1	Chloroform	<1	n-Propylbenzene	<1
Chloromethane	<1	Styrene	<1	2 & 4 Chlorotoluene	<1
1,1,2,2-Tetrachloroethane	<1	1,2-Dibromo-3-chloropropane	<1	Tetrachloroethane	<1
Dibromochloromethane	<1	Toluene	<1	1,2-Dibromoethane (EDB)	<1
1,2,3-Trichlorobenzene	<1	Dibromomethane	<1	1,2,4-Trichlorobenzene	<1
1,4-Dichlorobenzene	<1	1,1,2-Trichloroethane	<1	1,3-Dichlorobenzene	<1
1,1,1-Trichloroethane	<1	1,2-Dichloroethane	<1	Trichloroethene	<1
Dichlorodifluoromethane	<1	Trichlorofluoromethane	<1	1,2-Dichloroethene	<1
1,2,3-Trichloropropane	<1	1,1-Dichloroethane	<1	Bromochloromethane	<1
trans-1,2-Dichloroethene	<1	1,3,5-Trimethylbenzene	<1		
Vinyl Acetate	<1	1,1-Dichloromethane	<1		
Xylenes (total)	<5	1,2,4-Trimethylbenzene	<1		
Vinyl Chloride	<1	cis-1,2-Dichloroethene	<1		

In addition to the above analytes in Group 3, 40 mL and 60 mL vials are certified for:

Compound	Quantitation Limit (µg/L)
Total Organic Carbon	<600

Please keep this certificate for your records and to facilitate any necessary correspondence. If additional information is required, contact our Technical Service Department at (800) 443-1689.

Charles J. Willacker

Charles J. Willacker
Quality Assurance Manager

00017
1/01/27

PRODUCTION NUMBER: 337855

Item Number: 320-0125

Item Description: JAR SHORT CLEAR WM

Groups 1 and 2 are applicable

This is your Certificate of Analysis for I-CHEM Certified™ product which has been prepared in accordance with I-CHEM Performance-Based Specifications. This product meets or exceeds analyte specifications established in the U.S. EPA "Specification and Guidance for Contaminant-free Sample Containers" for use in Superfund and other hazardous waste programs.

Group 1. Glass and HDPE Sample containers for use in the analysis of Metals							
Analyte	Detection Limit (µg/L)	Analyte	Detection Limit (µg/L)	Analyte	Detection Limit (µg/L)	Analyte	Detection Limit (µg/L)
Aluminum	<80	Calcium (all HDPE)	<100	Magnesium	<100	Selenium	<2
Antimony	<5	Chromium	<10	Manganese	<10	Silver	<5
Arsenic	<2	Cobalt	<10	Mercury	<0.2	Sodium	<5000
Barium	<20	Copper	<10	Nickel	<20	Sodium (all HDPE)	<100
Beryllium	<0.5	Iron	<50	Potassium	<750	Thallium	<5
Cadmium	<1	Lead	<2	Potassium (all HDPE)	<100	Vanadium	<10
Calcium	<500					Zinc	<10

In addition to the above analytes, NALGENE® containers are certified for these analytes:							
Analyte	Detection Limit (µg/L)	Analyte	Detection Limit (µg/L)	Analyte	Detection Limit (µg/L)	Analyte	Detection Limit (µg/L)
Chloride	<100	Fluoride	<20	Nitrite	<50	Sulfate	<100
Cyanide	<10	Nitrite	<20	Formic acid (amber only)	<0.4	Sulfide	<30
Diquat (amber only)	<1.0					Sulfite	<1000

Group 2. Glass Sample Containers for use in the analysis of Semivolatiles and Pesticides/PCBs					
Compound	Quantitation Limit (µg/L)	Compound	Quantitation Limit (µg/L)	Compound	Quantitation Limit (µg/L)
Acenaphthene	<5	Acenaphthylene	<5	Anthracene	<5
Benzo(a)anthracene	<5	Benzo(a)pyrene	<5	Benzo(b)fluoranthene	<5
Benzo(k)fluoranthene	<5	Benzo(g,h,i)perylene	<5	Benzoic Acid	<20
Benzyl Alcohol	<5	4-Bromophenyl-phenylether	<5	Butylbenzylphthalate	<5
4-Chloroaniline	<5	4-Chloro-3-methylphenol	<5	bis-(2-Chloroethoxy)methane	<5
bis-(2-Chloroethyl)ether	<5	bis-(2-Chloroisopropyl)ether	<5	2-Chloronaphthalene	<5
2-Chlorophenol	<5	4-Chlorophenyl-phenylether	<5	Chrysene	<5
Di-n-butylphthalate	<5	Di-n-octylphthalate	<5	Dibenz(a,h)anthracene	<5
Dibenzofuran	<5	1,2-Dichlorobenzene	<5	1,4-Dichlorobenzene	<5
1,3-Dichlorobenzene	<5	3,3'-Dichlorobenzidine	<5	2,4-Dichlorophenol	<5
Diethylphthalate	<5	Dimethylphthalate	<5	2,4-Dinitrotoluene	<5
4,6-Dinitro-2-methylphenol	<20	2,4-Dinitrophenol	<20	Fluoranthene	<5
2,6-Dinitrotoluene	<5	bis-(2-Ethylhexyl)phthalate	<5	Hexachlorobutadiene	<5
Fluorene	<5	Hexachlorobenzene	<5	Indeno(1,2,3-cd)pyrene	<5
Hexachlorocyclopentadiene	<5	Hexachloroethane	<5	2-Methylphenol	<5
Isophorone	<5	2-Methylnaphthalene	<5	4-Nitroaniline	<20
4-Methylphenol	<5	2-Nitroaniline	<20	N-Nitrosodimethylamine	<5
4-Nitroaniline	<20	N-Nitroso-di-n-propylamine	<5	Nitrobenzene	<5
N-Nitrosodiphenylamine	<5	Naphthalene	<5	Pentachlorophenol	<20
2-Nitrophenol	<5	4-Nitrophenol	<20	Pyrene	<5
Phenanthrene	<5	Phenol	<5	2,4,6-Trichlorophenol	<5
1,2,4-Trichlorobenzene	<5	2,4,5-Trichlorophenol	<20	Aldrin	<0.01
Azobenzene	<5	Carbazole	<5	Alpha-BHC	<0.01
4,4-DDD	<0.02	Endosulfan II	<0.02	Beta-BHC	<0.01
4,4-DDE	<0.02	Endosulfan Sulfate	<0.02	Delta-BHC	<0.01
4,4-DDT	<0.02	Endrin	<0.02	Gamma-BHC	<0.01
Dieldrin	<0.02	Endrin Aldehyde	<0.02	Heptachlor Epoxide	<0.01
Endosulfan I	<0.01	Heptachlor	<0.01	Alpha-Chlordane	<0.01
Methoxychlor	<0.10	Endrin Ketone	<0.02	Aroclor-1016	<0.20
Gamma-Chlordane	<0.01	Toxaphene	<0.30	Aroclor-1242	<0.20
Aroclor-1221	<0.20	Aroclor-1232	<0.20	Aroclor-1260	<0.20
Aroclor-1248	<0.20	Aroclor-1254	<0.20		
Aroclor-1262	<0.20	Aroclor-1268	<0.20		

Group 3. Glass Sample Containers for use in the analysis of Volatiles					
Compound	Quantitation Limit (µg/L)	Compound	Quantitation Limit (µg/L)	Compound	Quantitation Limit (µg/L)
Acetone	<5	1,3-Dichloropropene	<1	Benzene	<1
2,2-Dichloropropene	<1	Bromobenzene	<1	1,2-Dichloropropane	<1
Bromodichloromethane	<1	trans-1,3-Dichloropropene	<1	Bromoforn	<1
cis-1,3-Dichloropropene	<1	Bromomethane	<1	1,1-Dichloropropene	<1
2-Butanone	<5	Ethylbenzene	<1	tert-Butylbenzene	<1
Hexachlorobutadiene	<1	sec-Butylbenzene	<1	2-Hexanone	<5
n-Butylbenzene	<1	Isopropylbenzene	<1	Carbon Disulfide	<1
p-Isopropyltoluene	<1	Carbon Tetrachloride	<1	4-Methyl-2-pentanone	<5
Chlorobenzene	<1	Methylene Chloride	<2	Chloroethane	<1
Naphthalene	<1	Chloroform	<1	n-Propylbenzene	<1
Chloromethane	<1	Styrene	<1	2 & 4 Chlorotoluene	<1
1,1,2,2-Tetrachloroethane	<1	1,3-Dibromo-3-chloropropene	<1	Tetrachloroethene	<1
Dibromochloromethane	<1	Toluene	<1	1,2-Dibromoethane (EDB)	<1
1,2,3-Trichlorobenzene	<1	Dibromomethane	<1	1,2,4-Trichlorobenzene	<1
1,4-Dichlorobenzene	<1	1,1,2-Trichloroethane	<1	1,3-Dichlorobenzene	<1
1,1,1-Trichloroethane	<1	1,2-Dichlorobenzene	<1	Trichloroethene	<1
Dichlorodifluoromethane	<1	Trichlorofluoromethane	<1	1,2-Dichloroethene	<1
1,2,3-Trichloropropene	<1	1,1-Dichloroethane	<1	Bromochloromethane	<1
trans-1,2-Dichloroethene	<1	1,3,5-Trimethylbenzene	<1		
Vinyl Acetate	<1	1,1-Dichloroethene	<1		
Xylenes (total)	<5	1,2,4-Trimethylbenzene	<1		
Vinyl Chloride	<1	cis-1,2-Dichloroethene	<1		

In addition to the above analytes in Group 3, 40 mL and 60 mL vials are certified for:

Compound	Quantitation Limit (µg/L)
Total Organic Carbon	<600

Please keep this certificate for your records and to facilitate any necessary correspondence. If additional information is required, contact our Technical Service Department at (800) 443-1689.

Charles J. Willacker
Charles J. Willacker
Quality Assurance Manager

90037
1/91 Z



PRODUCTION NUMBER: 344054



Item Number: 320-0250

Item Description: JAR SHORT CLEAR WM

Groups 1 and 2 are applicable

This is your Certificate of Analysis for I-CHEM Certified™ product which has been prepared in accordance with I-CHEM Performance-Based Specifications. This product meets or exceeds analyte specifications established in the U.S. EPA "Specification and Guidance for Contaminant-free Sample Containers" for use in Superfund and other hazardous waste programs.

Group 1. Glass and HDPE Sample containers for use in the analysis of Metals							
Analyte	Detection Limit (µg/L)	Analyte	Detection Limit (µg/L)	Analyte	Detection Limit (µg/L)	Analyte	Detection Limit (µg/L)
Aluminum	<80	Calcium (all HDPE)	<100	Magnesium	<100	Selenium	<2
Antimony	<5	Chromium	<10	Manganese	<10	Silver	<5
Arsenic	<2	Cobalt	<10	Mercury	<0.2	Sodium	<5000
Barium	<20	Copper	<10	Nickel	<20	Sodium (all HDPE)	<100
Beryllium	<0.5	Iron	<50	Potassium	<750	Thallium	<5
Cadmium	<1	Lead	<2	Potassium (all HDPE)	<100	Vanadium	<10
Calcium	<500					Zinc	<10

In addition to the above analytes, NALGENE® containers are certified for these analytes:

Analyte	Detection Limit (µg/L)	Analyte	Detection Limit (µg/L)	Analyte	Detection Limit (µg/L)	Analyte	Detection Limit (µg/L)
Chloride	<100	Fluoride	<20	Nitrite	<50	Sulfate	<100
Cyanide	<10	Nitrate	<20	Pamquat (amber only)	<0.4	Sulfide	<30
Diquat (amber only)	<1.0					Sulfite	<1000

Group 2. Glass Sample Containers for use in the analysis of Semivolatiles and Pesticides/PCBs							
Compound	Quantitation Limit (µg/L)	Compound	Quantitation Limit (µg/L)	Compound	Quantitation Limit (µg/L)	Compound	Quantitation Limit (µg/L)
Acenaphthene	<5	Acenaphthylene	<5	Anthracene	<5	Benzo(a)fluoranthene	<5
Benzo(a)anthracene	<5	Benzo(a)pyrene	<5	Benzoic Acid	<20	Butylbenzophthalate	<5
Benzo(k)fluoranthene	<5	Benzo(g,h,i)perylene	<5	Bis(2-ethylhexyl)phthalate	<5	Chloroethoxymethane	<5
Benzyl Alcohol	<5	4-Bromophenyl-phenylether	<5	2-Chloronaphthalene	<5	Chrysene	<5
4-Chloroaniline	<5	4-Chloro-3-methylphenol	<5	Chrysene	<5	Dibenz(a,h)anthracene	<5
Bis(2-Chloroethyl)ether	<5	bis-(2-Chloroisopropyl)ether	<5	1,4-Dichlorobenzene	<5	1,4-Dichlorophenol	<5
2-Chlorophenol	<5	4-Chlorophenyl-phenylether	<5	2,4-Dichlorophenol	<5	2,4-Dinitrotoluene	<5
Di-n-butylphthalate	<5	Di-n-octylphthalate	<5	Fluoranthene	<5	Hexachlorobutadiene	<5
Dibenzofuran	<5	1,2-Dichlorobenzene	<5	Hexachlorocyclopentadiene	<5	Hexachlorocyclopentadiene	<5
1,3-Dichlorobenzene	<5	3,3'-Dichlorobenzidine	<5	Isophthalene	<5	Hexachlorocyclopentadiene	<5
Diethylphthalate	<5	Dimethylphthalate	<5	4-Methylphenol	<5	Indeno(1,2,3-cd)pyrene	<5
4,6-Dinitro-2-methylphenol	<20	2,4-Dinitrophenol	<20	4-Nitrophenol	<5	2-Methylphenol	<5
2,6-Dinitrotoluene	<5	bis-(2-Ethylhexyl)phthalate	<5	Phenol	<5	3-Nitroaniline	<20
Fluorene	<5	Hexachlorobenzene	<5	Naphthalene	<5	N-Nitrosodimethylamine	<5
Hexachlorocyclopentadiene	<5	Hexachloroethane	<5	4-Nitrophenol	<20	Nitrobenzene	<5
Isophthalene	<5	2-Methylnaphthalene	<5	Phenol	<5	Pentachlorophenol	<20
4-Methylphenol	<5	2-Nitroaniline	<20	2,4,5-Trichlorophenol	<20	Pyrene	<5
4-Nitroaniline	<20	N-Nitroso-di-n-propylamine	<5	Carbazole	<5	2,4,6-Trichlorophenol	<5
N-Nitrosodiphenylamine	<5	Naphthalene	<5	Endosulfon II	<0.02	Aldrin	<0.01
2-Nitrophenol	<5	4-Nitrophenol	<20	Endosulfon Sulfate	<0.02	Alpha-BHC	<0.01
Phenanthrene	<5	Phenol	<5	Endrin	<0.02	Beta-BHC	<0.01
1,2,4-Trichlorobenzene	<5	2,4,5-Trichlorophenol	<20	Endrin Aldehyde	<0.02	Delta-BHC	<0.01
Azobenzene	<5	Carbazole	<5	Heptachlor	<0.01	Gamma-BHC	<0.01
4,4-DDD	<0.02	Endosulfon II	<0.02	Heptachlor Epoxide	<0.01	Heptachlor Epoxide	<0.01
4,4-DDE	<0.02	Endosulfon Sulfate	<0.02	Alpha-Chlordane	<0.01	Alpha-Chlordane	<0.01
4,4-DDT	<0.02	Endrin	<0.02	Aroclor-1016	<0.20	Aroclor-1016	<0.20
Dieldrin	<0.02	Endrin Aldehyde	<0.02	Aroclor-1242	<0.20	Aroclor-1242	<0.20
Endosulfon I	<0.01	Heptachlor	<0.01	Aroclor-1254	<0.20	Aroclor-1254	<0.20
Methoxychlor	<0.10	Endrin Ketone	<0.02	Aroclor-1260	<0.20	Aroclor-1260	<0.20
Gamma-Chlordane	<0.01	Toxaphene	<0.30				
Aroclor-1221	<0.20	Aroclor-1222	<0.20				
Aroclor-1248	<0.20	Aroclor-1254	<0.20				
Aroclor-1262	<0.20	Aroclor-1268	<0.20				

Group 3. Glass Sample Containers for use in the analysis of Volatiles							
Compound	Quantitation Limit (µg/L)	Compound	Quantitation Limit (µg/L)	Compound	Quantitation Limit (µg/L)	Compound	Quantitation Limit (µg/L)
Acetone	<5	1,3-Dichloropropane	<1	Benzene	<1	Benzo(a)fluoranthene	<5
2,2-Dichloropropane	<1	Bromobenzene	<1	1,2-Dichloropropane	<1	Benzo(k)fluoranthene	<5
Bromodichloromethane	<1	trans-1,3-Dichloropropane	<1	Bromofuran	<1	Butylbenzophthalate	<5
cis-1,3-Dichloropropane	<1	Bromomethane	<1	1,1-Dichloropropane	<1	Chloroethoxymethane	<5
2-Butanone	<5	Ethylbenzene	<1	tert-Butylbenzene	<1	Chloroethane	<1
Hexachlorobutadiene	<1	sec-Butylbenzene	<1	2-Hexanone	<5	n-Propylbenzene	<1
n-Butylbenzene	<1	Isopropylbenzene	<1	Carbon Disulfide	<1	2,4,4-Chlorotoluene	<1
p-Isopropyltoluene	<1	Carbon Tetrachloride	<1	4-Methyl-2-pentanone	<5	Tetrachloroethene	<1
Chlorobenzene	<1	Methylene Chloride	<2	Chloroethane	<1	1,2-Dibromochloroethane (EDB)	<1
Naphthalene	<1	Chloroform	<1	n-Propylbenzene	<1	1,2,4-Trichlorobenzene	<1
Chloromethane	<1	Styrene	<1	2,4,4-Chlorotoluene	<1	1,3-Dichlorobenzene	<1
1,1,2,2-Tetrachloroethane	<1	1,2-Dibromo-3-chloropropane	<1	Tetrachloroethene	<1	Trichloroethene	<1
Dibromochloromethane	<1	Toluene	<1	1,2-Dichlorobenzene	<1	1,2-Dichloroethene	<1
1,2,3-Trichlorobenzene	<1	Dibromomethane	<1	Trichlorofluoromethane	<1	Bromochloromethane	<1
1,4-Dichlorobenzene	<1	1,1,2-Trichloroethane	<1	1,1-Dichloroethane	<1		
1,1,1-Trichloroethane	<1	1,3-Dichlorobenzene	<1	1,3,5-Trimethylbenzene	<1		
Dichlorodifluoromethane	<1	Trichlorofluoromethane	<1	1,1-Dichloroethene	<1		
1,2,3-Trichloropropane	<1	1,1-Dichloroethane	<1	1,2,4-Trimethylbenzene	<1		
trans-1,2-Dichloroethene	<1	1,1-Dichloroethene	<1	cis-1,2-Dichloroethene	<1		
Vinyl Acetate	<1						
Xylenes (total)	<5						
Vinyl Chloride	<1						

In addition to the above analytes in Group 3, 40 mL and 60 mL vials are certified for:

Compound Quantitation Limit (µg/L)

Total Organic Carbon <600

Please keep this certificate for your records and to facilitate any necessary correspondence. If additional information is required, contact our Technical Service Department at (800) 443-1689.

Charles J. Willacker
Charles J. Willacker
Quality Assurance Manager

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International

I-CHEM
Brand Products

CERTIFICATE OF ANALYSIS

PRODUCTION NUMBER: 341175

Item Number: 320-0500

Item Description: JAR SHORT CLEAR WM

Groups 1 and 2 are applicable

This is your Certificate of Analysis for I-CHEM Certified™ product which has been prepared in accordance with I-CHEM Performance-Based Specifications. This product meets or exceeds analyte specifications established in the U.S. EPA "Specification and Guidance for Contaminant-free Sample Containers" for use in Superfund and other hazardous waste programs.

Group 1. Glass and HDPE Sample containers for use in the analysis of Metals							
Analyte	Detection Limit (µg/L)	Analyte	Detection Limit (µg/L)	Analyte	Detection Limit (µg/L)	Analyte	Detection Limit (µg/L)
Aluminum	<80	Calcium (all HDPE)	<100	Magnesium	<100	Selenium	<2
Antimony	<5	Chromium	<10	Manganese	<10	Silver	<5
Arsenic	<2	Cobalt	<10	Mercury	<0.2	Sodium	<5000
Boron	<20	Copper	<10	Nickel	<20	Sodium (all HDPE)	<100
Beryllium	<0.5	Iron	<50	Potassium	<750	Thallium	<5
Cadmium	<1	Lead	<2	Potassium (all HDPE)	<100	Vanadium	<10
Calcium	<500					Zinc	<10

In addition to the above analytes, NALGENE® containers are certified for these analytes:							
Analyte	Detection Limit (µg/L)	Analyte	Detection Limit (µg/L)	Analyte	Detection Limit (µg/L)	Analyte	Detection Limit (µg/L)
Chloride	<100	Fluoride	<20	Nitrite	<50	Sulfate	<100
Cyanide	<10	Nitrite	<20	Parquat (number only)	<0.4	Sulfide	<30
Diquat (amber only)	<1.0					Sulfite	<1000

Group 2. Glass Sample Containers for use in the analysis of Semivolatiles and Pesticides/PCBs							
Compound	Quantitation Limit (µg/L)	Compound	Quantitation Limit (µg/L)	Compound	Quantitation Limit (µg/L)	Compound	Quantitation Limit (µg/L)
Acenaphthene	<5	Acenaphthylene	<5	Anthracene	<5	Benzo(a)fluoranthene	<5
Benzo(a)anthracene	<5	Benzo(a)pyrene	<5	Benzo(b)fluoranthene	<5	Benzoic Acid	<20
Benzo(k)fluoranthene	<5	Benzo(g,h,i)perylene	<5	Butylbenzylphthalate	<5	Bis(2-chloroethoxy)methane	<5
Benzyl Alcohol	<5	4-Bromophenyl-phenylether	<5	Chrysene	<5	2-Chloronaphthalene	<5
4-Chloroaniline	<5	4-Chloro-3-methylphenol	<5	Dibenz(a,h)anthracene	<5	1,4-Dichlorobenzene	<5
bis-(2-Chloroethyl)ether	<5	bis-(2-Chloroisopropyl)ether	<5	1,4-Dichlorophenol	<5	2,4-Dichlorophenol	<5
2-Chlorophenol	<5	4-Chlorophenyl-phenylether	<5	2,4-Dinitrotoluene	<5	Fluoranthene	<5
Di-n-butylphthalate	<5	Di-n-octylphthalate	<5	Hexachlorobutadiene	<5	Indeno(1,2,3-cd)pyrene	<5
Dibenzofuran	<5	1,2-Dichlorobenzene	<5	2-Methylphenol	<5	3-Nitroaniline	<20
1,3-Dichlorobenzene	<5	3,3'-Dichlorobenzidine	<5	Nitrobenzene	<5	N-Nitrosodimethylamine	<5
Diethylphthalate	<5	Dimethylphthalate	<5	Pentachlorophenol	<20	Nitrobenzene	<5
4,6-Dinitro-2-methylphenol	<20	2,4-Dinitrophenol	<20	Pyrene	<5	2,4,6-Trichlorophenol	<5
2,6-Dinitrotoluene	<5	bis-(2-Ethylhexyl)phthalate	<5	Aldrin	<0.01	Alpha-BHC	<0.01
Fluorene	<5	Hexachlorobenzene	<5	Beta-BHC	<0.01	Delta-BHC	<0.01
Hexachlorocyclopentadiene	<5	Hexachloroethane	<5	Gamma-BHC	<0.01	Heptachlor Epoxide	<0.01
Isophorone	<5	2-Methylnaphthalene	<5	Heptachlor Epoxide	<0.01	Alpha-Chlordane	<0.01
4-Methylphenol	<5	2-Nitroaniline	<20	Alpha-Chlordane	<0.01	Aroclor-1016	<0.20
4-Nitroaniline	<20	N-Nitroso-di-n-propylamine	<5	Aroclor-1242	<0.20	Aroclor-1260	<0.20
N-Nitrosodiphenylamine	<5	Naphthalene	<5				
2-Nitrophenol	<5	4-Nitrophenol	<20				
Phenanthrene	<5	Phenol	<5				
1,2,4-Trichlorobenzene	<5	2,4,5-Trichlorophenol	<20				
Azobenzene	<5	Carbazole	<5				
4,4-DDD	<0.02	Endosulfan II	<0.02				
4,4-DDE	<0.02	Endosulfan Sulfate	<0.02				
4,4-DDT	<0.02	Endrin	<0.02				
Dieldrin	<0.02	Endrin Aldehyde	<0.02				
Endosulfan I	<0.01	Heptachlor	<0.01				
Methoxychlor	<0.10	Endrin Ketone	<0.02				
Gamma-Chlordane	<0.01	Toxaphene	<0.30				
Aroclor-1221	<0.20	Aroclor-1232	<0.20				
Aroclor-1248	<0.20	Aroclor-1254	<0.20				
Aroclor-1262	<0.20	Aroclor-1268	<0.20				

Group 3. Glass Sample Containers for use in the analysis of Volatiles							
Compound	Quantitation Limit (µg/L)	Compound	Quantitation Limit (µg/L)	Compound	Quantitation Limit (µg/L)	Compound	Quantitation Limit (µg/L)
Acetone	<5	1,3-Dichloropropene	<1	Benzene	<1	1,2-Dichloropropene	<1
2,2-Dichloropropene	<1	Bromobenzene	<1	Bromobenzene	<1	Bromobenzene	<1
Bromodichloromethane	<1	trans-1,3-Dichloropropene	<1	Bromobenzene	<1	Bromobenzene	<1
cis-1,3-Dichloropropene	<1	Bromomethane	<1	Bromobenzene	<1	Bromobenzene	<1
2-Butanone	<5	Ethylbenzene	<1	Bromobenzene	<1	Bromobenzene	<1
Hexachlorobutadiene	<1	sec-Butylbenzene	<1	Bromobenzene	<1	Bromobenzene	<1
n-Butylbenzene	<1	Isopropylbenzene	<1	Bromobenzene	<1	Bromobenzene	<1
p-Isopropyltoluene	<1	Carbon Tetrachloride	<1	Bromobenzene	<1	Bromobenzene	<1
Chlorobenzene	<1	Methylene Chloride	<2	Bromobenzene	<1	Bromobenzene	<1
Naphthalene	<1	Chloroform	<1	Bromobenzene	<1	Bromobenzene	<1
Chloromethane	<1	Styrene	<1	Bromobenzene	<1	Bromobenzene	<1
1,1,2,2-Tetrachloroethane	<1	1,3-Dibromo-3-chloropropene	<1	Bromobenzene	<1	Bromobenzene	<1
Dibromochloromethane	<1	Toluene	<1	Bromobenzene	<1	Bromobenzene	<1
1,2,3-Trichlorobenzene	<1	Dibromomethane	<1	Bromobenzene	<1	Bromobenzene	<1
1,4-Dichlorobenzene	<1	1,1,2-Trichloroethane	<1	Bromobenzene	<1	Bromobenzene	<1
1,1,1-Trichloroethane	<1	1,2-Dichlorobenzene	<1	Bromobenzene	<1	Bromobenzene	<1
Dichlorodifluoromethane	<1	Trichlorofluoromethane	<1	Bromobenzene	<1	Bromobenzene	<1
1,2,3-Trichloropropene	<1	1,1-Dichloroethane	<1	Bromobenzene	<1	Bromobenzene	<1
trans-1,2-Dichloroethene	<1	1,3,5-Trimethylbenzene	<1	Bromobenzene	<1	Bromobenzene	<1
Vinyl Acetate	<1	1,1-Dichloroethene	<1	Bromobenzene	<1	Bromobenzene	<1
Xylenes (total)	<5	1,2,4-Trimethylbenzene	<1	Bromobenzene	<1	Bromobenzene	<1
Vinyl Chloride	<1	cis-1,2-Dichloroethene	<1	Bromobenzene	<1	Bromobenzene	<1

In addition to the above analytes in Group 3, 40 mL and 60 mL vials are certified for:

Compound Quantitation Limit (µg/L)

Total Organic Carbon <600

Please keep this certificate for your records and to facilitate any necessary correspondence.

If additional information is required, contact our Technical Service Department at (800) 443-1689.

Charles J. Willacker

Charles J. Willacker
Quality Assurance Manager

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Appendix E.4

Chain of Custody and Sample Receipt Forms

142489

CHAIN OF CUSTODY

PROJECT # 99057 OUTSIDE LAB REQUIRED (Y/N) Y SAMPLE DATE 10/20/99
 CLIENT/LOCATION Air Products @ Stockton Coon, Stockton, CA PROJECT MANAGER EMM
 SAMPLE LOCATION Baghouse Inlet & Outlet METHOD(S) Ontario / Hydro TECHNICIAN JP & PG
 COMPLIANCE TEST (Y/N) N/A DATE DUE _____

DATE	TIME	TEST #	SAMPLE DESCRIPTION	CONTAINERS	SAMPLER	COMMENTS
10/20	:	1-Hg-I	Container 1, Filter	1	JP	-1
10/20	:	1-Hg-I	Container 2, Front Half Rinse	1	JP	-2
10/20	:	1-Hg-I	Container 3, Imp 1-3 & rinses	1	JP	-3
10/20	:	1-Hg-I	Container 4, Imp 4 & Rinse	1	JP	-4
10/20	:	1-Hg-I	Container 5, Imp 5-7 & rinses	1	JP	-5
/	:		!			
10/20	:	1-Hg-O	containers 1-5	5	PG	-6, 7, 8, 9, 10
10/21	:	2-Hg-I	containers 1-5	5	JP	-11, 12, 13, 14, 15
10/21	:	2-Hg-O	containers 1-5	5	PG	-16, 17, 18, 19, 20
10/22	:	3-Hg-I	containers 1-5	5	JP	-21, 22, 23, 24, 25
10/22	:	3-Hg-O	containers 1-5	5	PG	-26, -27, -28, -29, -30
/	:					
/	:					
/	:					
/	:					

TRANSFER OF SAMPLES FROM FIELD SAMPLE CUSTODIAN (FSC):

RELEASED BY (FSC)	DATE AND TIME	RECEIVED BY	DATE AND TIME
<u>D. Duncan</u>	<u>10/25/99 14:35</u>	<u>Lisa Pennell</u>	<u>10/25/99 2:40</u>
/	/ :	-	/ / :
/	/ :		/ / :

ANALYSIS REQUIRED: _____

142489

CHAIN OF CUSTODY

PROJECT # 99057 OUTSIDE LAB REQUIRED (Y/N) Y SAMPLE DATE 10/25/99
 CLIENT/LOCATION Air Products @ Stockton (Ogen, Stockton, CA) PROJECT MANAGER EMM
 SAMPLE LOCATION <Field Blanks> METHOD(S) Ontario/Hydro TECHNICIAN DD, PG, JP, KIC
 COMPLIANCE TEST (Y/N) N/A DATE DUE _____

DATE	TIME	TEST #	SAMPLE DESCRIPTION	CONTAINERS	SAMPLER	COMMENTS
10/21	:	FB-Hg-I	Container 1, Filter	1	JP	-31
10/21	:	FB-Hg-I	Container 2, Front Half Rinse	1		-32
10/21	:	FB-Hg-I	Container 3, Imp 1-3 & rinses	1		-33
10/21	:	FB-Hg-I	Container 4, Imp 4 & rinse	1		-34
10/21	:	FB-Hg-I	Container 5, Imp 5-7 & rinses	1	JP	-35
/	:		?			
10/22	:	FB-Hg-I	containers 1-5	5	PG	-36, 37, 38, 39, 40
/	:					
/	:					
/	:					
/	:					
/	:					
/	:					
/	:					
/	:					
/	:					

TRANSFER OF SAMPLES FROM FIELD SAMPLE CUSTODIAN (FSC):

RELEASED BY (FSC)	DATE AND TIME	RECEIVED BY	DATE AND TIME
<u>D. Dimoon</u>	<u>10/25/99 14:35</u>	<u>Chris Bennett</u>	<u>10/25/99 2:40</u>
	/ / :		/ / :
	/ / :		/ / :

ANALYSIS REQUIRED: _____

CERTIFICATION OF SAMPLE RECEIPT

PROJECT # 99057 OUTSIDE LAB REQUIRED (Y/N) Y SAMPLE DATE 10/20/99
 CLIENT/LOCATION Air Products @ Stockton, CA PROJECT MANAGER EMM
 SAMPLE LOCATION Baghouse Inlet & Outlet METHOD(S) Ontario/Hydro TECHNICIAN PM, JP, KJC
 COMPLIANCE TEST (Y/N) N/A DATE DUE _____

TEST #	COMPLETE DESCRIPTION
RB-Hg, 10/20/99	Reagent Blanks for 10/20/99, containers 7-12
RB-Hg, 10/21/99	" " " 10/21/99 " "
RB-Hg, 10/22/99	" " " 10/22/99 " "
FB-Hg-I	Inlet Field Blank, containers 1-5
FB-Hg-O	Outlet Field Blank, " "
1-Hg-I	Inlet Run 1, containers 1-5
1-Hg-O	Outlet " " " "
2-Hg-I	Inlet Run 2, " "
2-Hg-O	Outlet " " " "
3-Hg-I	Inlet Run 3, " "
3-Hg-O	Outlet " " " "

CHAIN OF CUSTODY PRIOR TO SHIPMENT:

RELEASED BY	DATE	TIME	RECEIVED BY	DATE	TIME
<u>D. Duncan</u>	<u>10/25/99</u>	<u>14:35</u>	<u>Je</u>	<u>10/25/99</u>	<u>:</u>
	<u>/ /</u>	<u>:</u>		<u>/ /</u>	<u>:</u>
	<u>/ /</u>	<u>:</u>		<u>/ /</u>	<u>:</u>

OUTSIDE LAB INFORMATION

SAMPLES SHIPPED TO Curtis & Tompkins
2323 F. Ave Street
Berkeley, CA 94710
 PHONE: (510) 486-0900

Avogadro SHIPPER Dan Duncan
 DATE 10/25/99
 CARRIER # N/A, carried by Dan Duncan
 AIR BILL # N/A
 RECIPIENT Joan Bennett
 COMPANY C&T
 DATE 10/25/99 2:40



142489

CHAIN OF CUSTODY

PROJECT # 99057 OUTSIDE LAB REQUIRED (Y/N) Y SAMPLE DATE 10/20/99
 CLIENT/LOCATION Air Products @ Stockton Co-op, Stockton, CA PROJECT MANAGER EMM
 SAMPLE LOCATION <Reagent Blanks> METHOD(S) Entario/hydro TECHNICIAN DICD
 COMPLIANCE TEST (Y/N) N/A DATE DUE _____

DATE	TIME	TEST #	SAMPLE DESCRIPTION	CONTAINERS	SAMPLER	COMMENTS
10/20	:	<u>Reagent Blank</u>	Container 7, 0.1N HNO ₃	1	DICD	-41
/	:		Container 8, 1N KCl	1		-42
/	:		Container 9, 5% HNO ₃ /10% H ₂ O ₂	1		-43
/	:		Container 10, H ₂ SO ₄ /KMnO ₄	1		-44
/	:		Container 11, Hydroxylamine Sulfate	1	✓	-45
10/20	:		Container 12, Filter	1	DICD	-46
/	:		:			
10/21	:	<u>Reagent Blank</u>	containers 7-12	6	DICD	-47, 48, 49, 50, 51, 52
10/22	:	<u>Reagent Blank</u>	containers 7-12	6	DICD	-53, 54, 55, 56, 57, 58
/	:					
/	:					
/	:					
/	:					
/	:					
/	:					
/	:					

TRANSFER OF SAMPLES FROM FIELD SAMPLE CUSTODIAN (FSC):

RELEASED BY (FSC)	DATE AND TIME	RECEIVED BY	DATE AND TIME
<u>[Signature]</u>	10/25/99 14:35	<u>[Signature]</u>	10/25/99 2:40
	/ / :		/ / :
	/ / :		/ / :

ANALYSIS REQUIRED: _____
